

# THE CHEMISTRY OF GAS MANUFACTURE

A PRACTICAL MANUAL

FOR THE USE OF

*GAS ENGINEERS, GAS MANAGERS, AND STUDENTS*

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With Coloured Plate and Numerous Illustrations



LONDON  
CROSBY LOCKWOOD AND SON  
7 STATIONERS' HALL COURT, LUDGATE HILL

1907

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## PREFACE.

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IN the present volume—which may be described as a small work upon an inexhaustible subject—the Author's aim has been to confine the work strictly to certain aspects of the Chemistry of Gas Manufacture, leaving the operations of manufacture alone.

A knowledge of the elementary truths and processes of Chemistry being pre-supposed, he has endeavoured to furnish the working gas engineer and manager with a concise manual, covering questions and points requiring attention in the ordinary course of his duties, which, it is believed, will be found of practical utility, especially in those gas works where the operations are not of so great an extent as to necessitate the employment of a separate chemical staff.

The volume, it is hoped, should also prove of utility to students of Gas Manufacture as well as to managers, as preparatory to the study of works of a larger scope.

For convenience of reference, the several subjects specifically treated of—including Coal and its characteristics, Furnace Gases, Products of Carbonisation, Materials for Purification, Fire-Bricks and Fire Clay, Photometry and Gas Testing, Carburetted Water-Gas, &c.—are dealt with in separate chapters (see "Contents"); while in the Appendices are given carefully selected extracts from matter previously published elsewhere, including the official regulations and instructions for testing coal-gas for illuminating power, calorific value and impurities, useful tables, data, statistics, &c.

For some of the illustrations the Author is indebted to the kindness of manufacturers of the apparatus illustrated, and he takes this opportunity of expressing his thanks.

For any practical suggestion for the improvement of the work with which readers thereof, or any of his professional brethren, may be disposed to favour him, the Author will be sincerely grateful: and all such communications will have his very careful consideration with a view to future editions.

HAROLD M. ROYLE.

GAS WORKS,  
BECKTON, *September 1907.*



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# THE CHEMISTRY OF GAS MANUFACTURE.

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## CHAPTER I.

### *PREPARATION OF STANDARD SOLUTIONS.*

THE most important part in analytical chemistry is the "making up" and the standardising of standard solutions. The most generally used of standard solutions are given here, and how to make them. Their method of use is explained in their respective places.

In analytical volumetric chemistry standard solutions are designated normal solutions, when they are of such a strength that 1 litre contains a weight of the reagent in grams equal to the chemical equivalent of that reagent; thus normal caustic soda contains 40 grams of NaOH per litre. The letter N is employed to denote standard normal solutions.

5N = 5 times the normal strength.

$\frac{N}{10}$  = one-tenth the normal strength.

&c. &c.

The following table gives the combining weight of a few of the most frequent reagents used in volumetric analysis:—

Name of Reagent.	Symbol.	Molecular Weight.	Combining Weight or Hydrogen Equivalent.
Ammonia - - -	$\text{NH}_3$	17.0	17.0
Barium hydrate - -	$\text{BaO}, \text{H}_2\text{O}$	171.0	85.5
„ carbonate - - -	$\text{BaCO}_3$	197.0	98.5
Calcium hydrate - -	$\text{CaO}, \text{H}_2\text{O}$	74.0	37.0
„ oxide - - -	$\text{CaO}$	56.0	28.0
„ carbonate - - -	$\text{CaCO}_3$	100.0	50.0
Iodine - - -	$\text{I}$	127.0	127.0
Potassium hydrate -	$\text{KHO}$	56.0	56.0
Sodium hydrate - -	$\text{NaOH}$	40.0	40.0
Hydrochloric acid -	$\text{HCl}$	36.5	36.5
Nitric acid - - -	$\text{HNO}_3$	63.0	63.0
Oxalic acid - - -	$\text{H}_2\text{C}_2\text{O}_4, 2\text{H}_2\text{O}$	126.0	63.0
Sulphuric acid - -	$\text{H}_2\text{SO}_4$	98.0	49.0

It will be observed from the above table that in the case of univalent substances, such as ammonia, iodine, sodium hydrate, &c., the equivalent and the atomic (or molecular, in case of salts) weight are the same, and in the case of bivalent substances, such as barium hydrate, barium carbonate, &c., the equivalent weight is one-half of the atomic weight (or molecular), and in the case of trivalent substances the equivalent weight would be one-third of the atomic weight.

### Indicators used in Volumetric Analysis.

*Cochineal Solution.*—This indicator is prepared by digesting a certain quantity of the powdered or bruised cochineal for several hours at a gentle heat in a solution of weak spirit, composed of 200 c.c. of methylated spirit and 600 c.c. of water. The substance is allowed to settle, and when clear it is decanted or filtered off, and is then ready for use.

Its normal colour is yellow, and this is changed to a reddish violet by alkalis; this reddish-violet colour is changed to yellow by mineral acid. But this reaction is slower in the case of weak organic acid. It should not

be d in the presence of compounds of iron, aluminium, or : tate.

is usually adopted for estimation of ammonia in the Re e's test, or in the estimation of ammonia in gas liqu and sulphate. This indicator is not affected by  $\text{CO}_2$ .

*thyl Orange*.—This indicator is prepared by dissolv ; some solid methyl orange in a small quantity of met lated spirit and diluting with water.

thyl orange is employed for the estimation of free am nia in gas liquor.

thyl orange is unaffected by  $\text{CO}_2$ , and is specially ada d for the titration of alkaline carbonates with mineral acic *vice versa*.

*molphtalein*.—This indicator is prepared by dissolv a little of the solid substance in alcohol. The solu n is colourless, but on the addition of an alkali it beco es a deep red colour. The colour is immediately char d when the liquid is acidified either with mineral or orga : acid.

cannot be employed in cases where carbonic acid is evol l, as carbonic acid destroys the colour, but as the bica nates (or acid carbonates) do not give the red colour with is compound, it is most valuable in indicating the first ge in the neutralisation of a normal carbonate, viz., the nversion of the normal into the acid carbonate. Phe: phtalein is not used in presence of ammonia.

*nus Solution*.—The solid litmus is boiled with hot water filter, and add a slight excess of acetic acid. The solu is evaporated until it becomes pasty, when an excess of n ylated spirit is added. The spirit precipitates the blue louring matter; the red colouring matter, together with e alkaline acetates, remains in solution. The blue prec ate is filtered and well washed with spirit. The pure ue colouring matter thus obtained is dissolved in warr water, and the solution is ready for use. As this solut loses its colour if not exposed to the air, it is only light covered so as to exclude dust, &c. A few drops of

chloroform well shaken up with it will prevent the formation of mould.

The solution is turned from blue to red by acid, and *vice versa* by alkalis: it cannot be used in the presence of  $\text{CO}_2$  unless the liquid is first of all boiled, and all the  $\text{CO}_2$  eliminated.

*Lucanid*.—This indicator is made by dissolving some of the solid in a weak solution of alcohol. It turns from brownish yellow to green on excess of alkali.

Used in Coleman & Smith test for naphthalene. Will not act properly in the presence of carbolic acid, which masks the point of neutralisation.

*Sodium Carbonate*,  $\text{Na}_2\text{CO}_3$ .—53 grams of  $\text{Na}_2\text{CO}_3$  per litre = N solution. This salt must be in an absolute state of purity, and is prepared by heating the purest sodium bicarbonate to a dull red heat for about ten to fifteen minutes, or until no further loss of carbon dioxide or water takes place. The salt must not be allowed to fuse. It is then cooled in a desiccator and weighed. To ensure that the decomposition is complete, it is again heated to a dull red heat for ten minutes, and after cooling in desiccator, it should weigh the same as before. 53 grams of this are now weighed out and dissolved in 1 litre of water.

This solution is used to standardise the acid solution by, so it is necessary to take very great care in its preparation.

*Ammonia*,  $\text{NH}_4\text{OH}$ .—The strong solution, sp. gr. .880 = 20N. This is diluted to any suitable strength that may be required.

*Ammonium Oxalate*,  $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ .—80 grams of salt in 1 litre of water = N solution.

*Ammonium Sulphate*,  $(\text{NH}_4)_2\text{SO}_4$ .—66 grams of salt in 1 litre of water = N solution.

*Ammonium Thiocyanate*,  $\text{NH}_4\text{CyS}$ .—76 grams of salt in 1 litre of water = N solution.

*Barium Hydroxide*,  $\text{Ba}(\text{HO})_2 \cdot 8\text{H}_2\text{O}$ .—31 grams in 1 litre of water =  $\frac{\text{N}}{5}$  solution.

# STANDARD SOLUTIONS

*Bromine Water, Br.*—Obtained by shaking excess of bromine with water until saturated.  $\frac{N}{10}$  solution.

*Calcium Hydroxide (Lime Water),  $\text{Ca}(\text{OH})_2$* —Obtained by shaking an excess of quicklime with water until saturated, filter =  $\frac{N}{20}$  solution roughly.

*Hydrochloric Acid.* Pure  $\text{HCl}$  is diluted with water until sp. gr. of 1.10 at 60 degrees Fahr. About 100 cc. acid are diluted to 1 litre by distilled water =  $\frac{N}{10}$  solution. Its exact strength is titrated against soda.

*Potassium Hydroxide,  $\text{KHO}$* —56 grams in 1 litre of water =  $\frac{N}{10}$  solution.

*Potassium Dichromate.* 4.903 grams in 1 litre of water =  $\frac{N}{10}$  solution, previously dried by gentle heating on a porcelain dish.

1 c.c. = .0056  $\text{Fe}$   
 1 c.c. = .0057  $\text{FeO}$   
 1 c.c. = .0086  $\text{Fe}_2\text{O}_3$   
 1 c.c. = .0089  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$   
 1 c.c. = .0107  $\text{Fe}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$

*Potassium Permanganate,  $\text{KMnO}_4$* —15.8 grams in 1 litre of water =  $\frac{N}{10}$  solution.

1 c.c. = .0056  $\text{Fe}$  &c. &c.

*Sodium Hydroxide,  $\text{NaOH}$* —40 grams in 1 litre of water =  $\frac{N}{10}$  solution.

*Sodium Thiosulphate,  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$* —24.8 grams in 1 litre of water =  $\frac{N}{10}$  solution.

*Iodine, I.*—12.7 grams per litre of water =  $\frac{N}{10}$  solution.

*Silver Nitrate,  $\text{AgNO}_3$* —16.976 grams in 1 litre of water =  $\frac{N}{10}$  solution.

1 c.c. = .01362 KCN grams

#### PREPARATION OF STANDARD SOLUTIONS.

For the estimation of chlorine in water, 4.79 grams in 1 litre of water

1 cc. will precipitate 1 milligram of Cl.

*Di-m. ortho-phosphate.*—This is prepared as follows:—  
2 lbs. of hydrogen disodium phosphate are dissolved in 1 gallon of water, and  $2\frac{1}{2}$  lbs. of cupric sulphate crystals in 1½ gallon of water. These solutions are well mixed and the resulting bright blue precipitate washed by decantation and then dried in a water bath at about 212 degrees Fahr. This material has a great affinity for  $\text{SH}_2$ .



## CHAPTER II

### COAL

THE word coal carries a large meaning to the geologist, and in the analysis required on a gasometer, it is not general to ascertain the percentage composition, but it is necessary and very important to analyse and ascertain the various benefits of one class of coal over another. It might be just as well to briefly mention the various stages in which woody or fibrous matter undergoes in its decomposition before it ultimately becomes coal.

When woody matter is buried the composition is greatly altered, with the liberation of hydrogen, marsh gas, and carbonic anhydride.

The more water there is present the more rapid is the decomposition.

Coal is the resultant product of this decay, under the prolonged influence of heat, moisture, and pressure.

The longer this influence has been at work the harder and richer in carbon is the coal.

Peat is the most recent product, and graphite the oldest or most prolonged.

The Table given here is by Prof. Raphael Meldola, F.R.S., and shows the gradual conversion of wood into the oldest carbonaceous material, graphite.

Name.	Carbon.	Hydrogen.	Oxygen.
I. Woody fibre (cellulose) -	50.0	6.0	44.0
II. Peat from Dartmoor -	54.0	5.2	28.2
III. Lignite or brown coal (an imperfectly carbonised vegetable deposit of more recent geological age than true coal) -	66.3	5.6	22.8
IV. Average bituminous coal -	77.0	5.0	11.2
V. Cannel coal from Wigan -	81.2	5.6	7.9
VI. Anthracite from Wales -	90.1	3.2	2.5
VII. Graphite - - - -	{ 94 to 99.5, the remainder being ash. The oldest carbonaceous material.		

The chief varieties of coal the gas chemist is called upon to deal with are as follows :—

(I.) Peat, which is hardly a coal in the true sense of the word, but which may at no distant date be used for gas-making purposes.

It is the first step in the decomposition of cellular fibre, and would in time become coal.

If at any time the price of coal was such as to prohibit its use, peat would then be brought into more notice, and would certainly be well worth consideration.

The one great drawback is the amount of water it contains, which is about 90 per cent. ; after it has been stored under cover for some considerable time the water is then decreased to about 60 per cent.

There are various patents on the market for decreasing this amount of water, but at present no great headway has been made in this country. On the Continent more has been done in this matter, and there are some very excellent continuous presses, which decrease the amount of water to a more reasonable figure.

In the carbonisation of dried peat the results are very similar to coal, giving a good yield of gas of from 10,000 to 11,000 cub. ft., and the purified gas test-

ing about sixteen candles on the No. 2 Metropolitan Argand.

The coke, however, is inferior, resembling the peat, and the tar and ammoniacal liquor are fair.

In December 1902 Mr John Miller, F.I.C., in the *Glasgow Herald*, gives some particulars of the peat industry on the Continent and in America, and urges that more attention ought to be given to the peat bogs of Scotland and Ireland. He says that from 1 ton of peat there can be produced 12,500 cub. ft. of gas free from sulphur, 16 lbs. acetic acid, 46 lbs. wood naphtha, 18 lbs. sulphate of ammonia, some tar, and 10½ lbs. of paraffin wax.

(2.) The next stage to be considered is lignite or brown coal, and this is peat in a more advanced stage of decomposition. It is not found in very large quantities in England and France, but large deposits occur on the Continent, and the yield of gas is very inferior.

(3.) The ordinary coal may be classified in two sections:—

(a.) Caking coals. (b.) Non-caking coals.

(a.) *Caking coals* are those that soften or fuse on heating, and on the expulsion of the volatile constituents leave a coke that has no cellular structure, or in any way resembles the shape of the original coal.

The exact cause of this caking is not known, as coals from the same district and of the same composition are not characteristic in this action, some caking, others not doing so.

The most obnoxious constituent of most caking coals is the sulphur, but this under the new Metropolitan Gas Act is not so serious as in the days of sulphur clauses.

(b.) *Non-caking coals*, when carbonised so that the volatile constituents are expelled, yield a coke which retains the original form of the coal, or else crumbles into small fragments, and as a fuel it is inferior to the coke of the caking coal variety. Non-caking coals do not differ much from caking coals in their elementary composition,

and the classification depends upon their behaviour on carbonising.

(4.) Cannel coals appear to hold an intermediary position between lignite and bituminous gas coals, and are richer in carbon, but poorer in oxygen (see Table).

The coke is obtained in the form of the cannel and is practically useless for fuel. It yields a very high quality gas, giving about 12,000 cub. ft. per ton of thirty candles gas or more. The best named varieties are Lesmahagow, Boghead, and Newbattle. The latter is the general class of cannel that is used, and its average result is as follows :—

Specific gravity	-	-	-	1.175 (water = 1,000).
Gas per ton at N.T.P.	-	-	-	13,720 cub. ft.
Specific gravity of gas	-	-	-	668 (air = 1,000).
Sperm value from 1 ton	-	-	-	1,708 lbs.
Illuminating power	-	-	-	35.24 candles.
Sulphuretted hydrogen (crude gas)	-	-	-	1.50 per cent., or 946.00 grains per 100 cub. ft.
Carbonic acid	-	-	-	3.00 per cent., or 2,451 grains per 100 cub. ft.
Carbonic oxide (CO)	-	-	-	8.00 per cent.
Coke per ton of coal	-	-	-	1,089.3 lbs., or 9.72 cwt.

This is a very rich cannel coal. The foul gas contains a rather large percentage of impurities.

The Lesmahagow cannel coal is practically exhausted, and very little is found on the market.

In the analysis required of a gas chemist by the engineer and manager, is not so much the percentage composition of the coal, but an approximate report on what he may expect from it when carbonising on the works. The usual report covers the following ground :—

- I. Yield of gas per ton of coal.
- II. Illuminating power.
- III. Coke per ton (total).
- IV. Coke per ton (saleable).
- V. Sperm value.

- VI. Carbonic acid ( $\text{CO}_2$ ).
- VII. Sulphuretted hydrogen ( $\text{SH}_2$ ).
- VIII. Sulphur in coal.
- IX. Sulphur in coke.
- X. Value of coke as a fuel, percentage of ash, &c.

The first seven items are carried out on an experimental plant, which is generally a small plant especially fitted up for this purpose, and is as follows:—

**Experimental Coal-Testing Plant.**—This plant consists of two small cast-iron retorts, 6 ft. in length, 6 in. wide, and 3 in. high, with a direct fired setting and damper to the flues, so that you are able to work one retort at the time, the second one being handy in case the other cracks. By means of the dampers one is able to keep the setting under perfect control. The retorts generally project a few inches from bed in place of the usual mouthpieces, and a 3-inch pipe is fitted for an ascension pipe which leads to the condensing tubes, which may be attached to an adjacent wall, and consist of ten tubes 10 ft. long and 3 in. in diameter, with proper screw caps on the top (for cleansing out in case of stoppages) and a small valve on the bottom for running off the condensed matter, as tar and liquid. It is unnecessary to have a washer, and the gas passes direct into the purifiers from the condensers.

The purifiers are two circular boxes, 24 in. diameter and 12 in. deep, fitted with three trays, on which the purifying material is spread.

Under the new gas regulation (see Appendix) it is only necessary to purify from sulphuretted hydrogen, the sulphur compounds being left in the gas, and the purifying material used is bog-ore or oxide of iron. Some lime can be used for experimental purposes, if necessary, on a coal which gives a very bad result owing to the  $\text{CO}_2$  being left in; this can be removed and the difference in the test reported on.

The gas from the outlet of purifiers is led direct to the experimental holder, which should hold about 20 cub. ft.

It is advisable to have two holders—one can be filled with gas from the retort and then blown into the other by means of weights and suitable connections. The gas can then be tested without having to wait for the gas to mix in the one holder. The gas can then be tested for illuminating power, calorific value, &c. &c. While this is being done, another charge can be put on, the gas going into the first holder. The charge is 2.24 lbs., or the one-thousandth part of a ton, and it is desirable to have a special weight made, or else take 2.25 lbs. and divide result by .995. The apparatus is shown in Fig. 1.

When the retort is at the desired heat, the charge is weighed out and placed in a small scoop made to fit the retort. The lid is smeared round with clay or "pug," the scoop is driven in, reversed, and withdrawn, and the door put on at once. It is necessary to put in a preliminary charge, to clean the apparatus of all air and previous samples of gas. The gas is blown away on the outlet of holder, or burnt at the mouthpiece, and directly the holder is around shut the cock on the inlet to holder, which must be opened directly the lid is put on after the fresh charge. The charge will take from thirty to forty minutes to burn off, and, as the plant is fitted with pressure gauges, by shutting off the inlet to holder the gauge will indicate whether gas is still being made. The holder is weighted in the first instance so as to give a level gauge at this point. To increase the make, increase the heats, and put a small vacuum on the retort.

The holders are connected up to a photometer, either a Letheby, or preferably a standard instrument as the Harcourt Table photometer. The results are then certainly more reliable.

Instead of wasting time waiting for the gas to mix in the one holder, blow into the second holder; this will save time and greatly facilitate the mixing. Another charge of the same coal can be put on, and be burning off, leaving the experimenter at liberty to test the first charge.

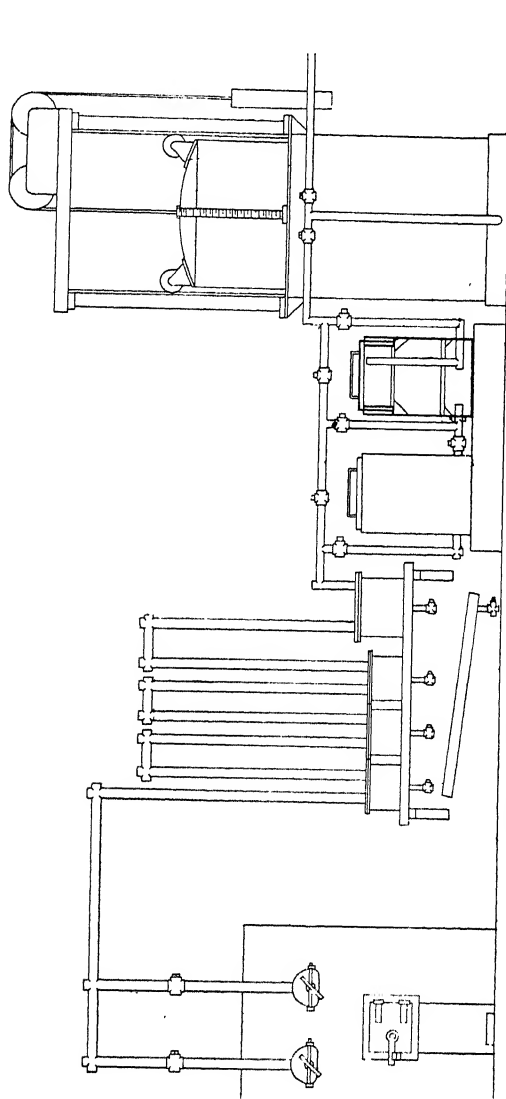


FIG. 1.—APPARATUS USED FOR EXPERIMENTALLY TESTING A SAMPLE OF COAL.

The illuminating power and calorific value is determined as per Metropolitan Referees' Notifications (see Appendix).

It is necessary to test at least four samples of the same coal, and take the average.

The tar and ammoniacal liquor are allowed to accumulate during the four charges, and are then run off from the bottom of the condensers, and are run into a trough and the average taken.

The coke is drawn from the retort into an iron tray and put on one side to cool; it is not quenched, and when cold is weighed and calculated to its amount per ton of coal carbonised.

A small piece from each charge is kept for further analysis in the laboratory.

It is sometimes necessary to ascertain the amount of impurities in the crude gas. In this case it is advisable to have a special holder, which can be connected up from the inlet of purifiers. A charge can be put in the retort as before, the gas passing through condensers only and going direct into holder. The holder can then be weighted and the gas tested for  $\text{CO}_2$  and  $\text{SH}_2$  as follows:—

A series of weighed U tubes filled with soda lime (four being sufficient) are connected up to one another by means of rubber tubing; this is connected on to three Woulfe bottles containing cadmium chloride which has been acidified with a few drops of hydrochloric acid, and the gas is then passed through these, going through a small experimental meter last—first soda lime, and then the cadmium chloride.

A foot to 2 feet is quite sufficient for a test. The tubes are disconnected and weighed; the increase in weight gives the direct amount of carbonic acid in the quantity of gas taken; this is calculated to grains per 100 cub. ft.

The Woulfe bottles are then washed out, and a little bromine water added; boil to expel excess of bromine, acidulate with hydrochloric acid, and add barium chloride; filter off the resultant precipitate of barium sulphate, dry



and burn off in a platinum crucible. The weight of  $\text{BaSO}_4$ , multiplied by 0.1459, gives  $\text{SH}_2$  in quantity of gas taken. This can easily be calculated to grains of  $\text{SH}_2$  per 100 cub. ft.

The working data and calculations for the above test are as follows:—

*Working data—1st Charge.*

Charge	-	-	-	-	2.24 lbs. coal.
Gas made	-	-	-	-	10.9 cub. ft.
Temperature	-	-	-	-	70 degrees.
Barometer	-	-	-	-	30.26.
Tabular number	-	-	-	-	.982.
Coke	-	-	-	-	24 oz.
Calorific value	-	-	-	-	610 B.T.U.
Illuminating power on table photometer					15.80 candles corrected.
Sperm	-	-	-	-	591 lbs. per ton.

These figures are worked out to the make per ton, and all four charges are done separately and average taken. The sperm value is calculated as follows:—

$$\frac{\text{I.P.} \times \text{yield per ton} \times 120}{5 \times 7000}$$

$$\text{or I.P.} \times \text{yield per ton} \times 0.00343.$$

*Example—*

$$\begin{array}{r}
 10,900 \text{ yield per ton.} \\
 15.8 \\
 \hline
 87200 \\
 54500 \\
 10900 \\
 \hline
 17222 \\
 .00343 \\
 \hline
 51666 \\
 68888 \\
 51666 \\
 \hline
 590.7146 \text{ lbs. sperm per ton.}
 \end{array}$$

The other calculations are simple; the working out of illuminating value and calorific value are given in their respective places or chapter.

*Example of Estimation of Carbonic Acid (CO<sub>2</sub>) and Sulphuretted Hydrogen (SH<sub>2</sub>):—*

*Carbonic Acid.*

	1st Tube.	2nd Tube.	3rd Tube.	4th Tube.
Before	- 1000.60	1010.80	1018.60	1030.1 grains.
After	- 1003.10	1010.90	1018.60	1030.1 „
	<hr/>	<hr/>	<hr/>	<hr/>
Increase	- 2.50	0.10	Nil	Nil

Gas passed, 1.20 cub. ft.

Temperature, 68 degrees.

Barometer, 30.26.

Tabular number, 987.

987)1.200(1.21 corrected gas used.

987

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2130

1974

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1560

1.21 cub. ft. has 2.1 grains CO<sub>2</sub>;

∴ 100 cub. ft. have  $2.1 \times 100 \div 1.21 = 2.1$

100

---

1.21)2100(181

121

---

.990

968

= 1818.18 grains CO<sub>2</sub> per 100 cub. ft.

*Estimation of Sulphuretted Hydrogen (SH<sub>2</sub>).—*Weight of BaSO<sub>4</sub> × 0.1459 = grains of sulphuretted hydrogen per 100 cub. ft.

## ANALYSIS OF COALS.

17

EXTRACTS ANALYSIS OF COALS, BY MR JAMES PATERSON (FROM "LITHOLOGY OF GAS COALS").

Name of Coal.	District.	Gas per ton.	Coke per ton.	Ash in Coke.	Specific Gravity of Coal.	Con- densa- tion by Bro- mine.	Illumi- nating Power.	Value of 1 cub. ft. in Grains of Sperm.	Weight of Gas per ton of Coal in lbs. of Sperm.	Gas Purified by 1 cwt. of Lime.	Sulphur per cent.
Aston Hall premier coal	Hawarden	11,250	1,368	3.37	1.264	4.00	16.17	388.00	623.43	10,000	...
Birley silkstone	Barnsley	12,450	1,463	1.68	1.266	4.50	16.27	390.48	688.78	15,000	...
Derbyshire silkstone	Rotherham	11,020	1,411	3.21	1.254	...	15.43	370.32	583.00	15,000	...
Hazelwood Coal and Cannel Co.	Mold	10,500	1,282	5.10	1.260	3.85	14.81	355.44	533.15	...	5.10
Lidgate Colliery	Carlisle	12,400	1,400	7.72	1.284	4.00	15.26	366.24	648.77	...	0.971
Mirfield Coal Co., coal and cannel mixed	Mirfield	12,600	1,430	1.88	1.252	5.15	16.67	400.00	720.00	17,000	...
Newton Coal and Iron Co., gas coal	Cheshire	10,400	1,320	4.10	1.261	4.25	16.48	396.00	588.34	...	1.153
Newton, Chamber, & Co.—											
1. Norfolk silkstone	Thorncliffe	13,300	1,331	0.79	1.252	4.25	15.14	395.36	694.18	17,500	...
2. Thin seam	"	12,500	1,336	1.32	1.255	4.00	14.27	342.48	611.57	15,500	...
3. Best silkstone	"	12,180	1,441	1.57	1.248	4.75	16.38	393.00	683.82	...	...
4. Screened gas coal	"	12,350	1,400	1.30	1.251	4.50	16.00	384.00	677.49	...	...
5. Silkstone soft	"	11,500	1,482	0.79	1.247	5.00	16.75	402.00	660.43	...	...
Pelton Main, Chester-le-Street, D'ham	Durham	11,600	1,560	3.25	1.282	4.75	17.66	424.00	702.62	...	1.101
Pope & Pearson—											
1. Screened silkstone	West Riding	12,750	1,368	2.44	1.274	4.25	15.26	366.24	667.00	16,000	...
2. Hard coals	"	10,900	1,368	7.31	1.283	4.00	15.50	372.00	579.26	15,000	...
Ravenhead upper delph	St Helens	10,425	1,305	4.70	1.290	...	15.61	374.64	557.04	...	0.738
Sheepbridge Coal and Iron Co.	Chesterfield	10,800	1,428	5.07	1.277	5.00	15.53	372.72	575.00	...	0.841
Staveley gas coal, top	Derbyshire	9,040	1,395	4.32	...	...	11.52	270.00	350.00	13,000	...
" bottom	"	10,140	1,340	4.50	1.280	...	12.23	294.00	424.00	13,000	...
Unston Colliery Co.	Sheffield	10,375	1,400	2.00	1.274	...	13.57	325.70	482.73	...	...
Wigan and Whiston gas coal	Wigan	9,750	1,412	2.94	1.289	3.50	13.75	330.00	459.64	...	...
Woodleford gas coal	Leeds	12,200	1,305	6.30	1.292	4.00	16.00	384.00	614.40	...	1.173

\* Illuminating power by No. 1 Metropolitan burner.

EXTRACT ANALYSIS OF COALS, FROM MR JAMES PATERSON  
(“PHYSIOLOGY OF GAS COAL”).

Description of Coal	N <sub>2</sub>	C	H <sub>2</sub>	N <sub>2</sub>	Sulphur	Oxygen	Ash	Water evaporated in Pounds from 1 lb. by 1100 F.		
								Adm.	theo.	Calori- fically retort house
								Test	Value	Test
Wigan Coals from										
Arley	1.2608	34.5	24.0	0.98	1.05	5.87	3.32	8.83	15.47	15.00
Lindsey Arley	1.2608	39.0	5.60	1.40	1.51	5.53	2.00	7.44	15.38	15.95
Haugh, coal	1.2608	2.20	5.47	1.25	1.48	5.64	3.00	7.00	15.47	14.54
" 5 feet	1.2607	4.21	5.03	0.77	2.00	8.09	9.21	7.21	13.77	13.20
Blackburn, Birtle dale	1.2608	2.01	5.55	1.08	1.43	5.28	4.05	8.81	15.50	...
Blackburn, "	1.2608	2.70	5.55	1.48	1.07	4.89	4.31	8.29	15.03	13.20
Rushy Park mine	1.2607	7.70	5.23	1.32	1.01	8.99	5.60	8.08	14.35	14.12
Blackburn, Rushy Park	1.2708	1.10	5.99	1.35	1.62	7.20	2.68	8.02	15.51	14.12
Lapack, Rushy Park	1.35	80.47	5.72	1.27	1.39	8.33	2.82	7.98	15.19	14.12
Wigan, Larnell	1.23	70.23	6.08	1.18	1.43	7.24	4.84	7.70	15.30	12.10

These figures are given to show the working methods, and how the results are obtained.

In these reports it is absolutely necessary to have a standard coal, either of some coal which has been worked in the retort house, and which one knows will give certain results; this coal can then be used on the experimental plant, and its result compared with the special coal under analysis. In this way one may have an absolute standard coal to go on, and you can give an absolute reliable opinion on any coal as to how it will behave under working conditions.

There have been a good many objections to the small results obtained on an experimental set, but if these coals are compared with any coal of which its characteristics are known, these objections fall to the ground.

In drawing out the report it is necessary to include this standard coal in the report, and give the result obtained in the experimental set and its result in the retort house. The engineer is then able to thoroughly understand the test, and due appreciation is given.

It is also necessary to use the same coke as fuel right through the test, and it is only by great attention to details that one arrives at a result which will be in any way confirmatory with the working result. If this attention is given there is not any reason on earth why the results obtained should not be trustworthy, and a great guide to the proper working of a coal.

The tabulated copy of a report on a coal is here given, and shows how the report is written out. In these analytical reports on coal there is only one exception that can be taken, *i.e.*, the coal is not carbonised by the heat derived from its own coke.

In the retort house, for instance, there is a good coal being carbonised which, besides giving a good make, &c., gives a coke which is an excellent fuel. Another class of coal is brought in which, with the good heats in the setting, gives a good make. After a few hours working this coal, and when the furnaces have been charged with the coke obtained from this coal, the heats begin to drop off, which, on investigation, is found to be due to the bad quality of the coke. This renders the heat in the retort less, and a corresponding drop in the make per ton is noticed.

This information can be practically gleaned from the analytical report if it is properly studied; for instance, the ash in the coke of the standard sample must be compared with that in the sample under examination, and also the water evaporated per lb. of fuel. It would be as well if the chemists drew attention to anything of this sort, so as to save the engineer time in going through too many details. Special attention should be taken of the ash, as if this is high one may expect bad heats, which would be due to the increase in the non-combustible matter which would clog the furnace and prevent the proper proportion of primary air, and would require more labour in "pricking up" the furnace to get the utmost efficiency out of this coke.

## COAL.

REPORT ON A SAMPLE OF TAG COAT.

[illegible]

\* Illuminating power by No. 2 Metropolitan burner.

### The Analysis of Coal and Coke in the Laboratory.

After ascertaining the qualities of the coal on the experimental basis it is necessary to examine both the coal and coke in the laboratory for moisture, sulphur, &c. These methods are as follows :—

(1.) **Moisture.**— It is often supposed that to obtain the moisture in coal is to weigh out a certain quantity and dry it in the water oven ; this is an erroneous method, and gives results that are, to say the least, not reliable. It is found with this method the coal decreases in weight to a certain point, and then increases again. This shows that this method is erroneous. The correct and proper method is to pass dry gas or air over the coal, and absorb the moisture that has been taken out from the coal by the dried air or gas in a tube filled with calcium chloride. A large cylinder is filled with calcium chloride, and four small U tubes with the same material ; these latter are weighed and the coal is weighed into a dried U tube, and weight taken and noted. (Fig. 2.) The method of procedure is :—The gas or air is passed through the large cylinder of calcium chloride, and then through two of the small U tubes, which are weighed before and after the test to ascertain that all the moisture is taken out ; the gas then passes through the tube which contains the coal, and which is suspended in a beaker of boiling water ; on the outlet of this tube is the other two weighed U tubes, which absorb the moisture in the gas which has been taken up from the coal.

The gas is burnt on the outlet, and the test is kept going for two to three hours, when it is disconnected, and the tubes on the outlet weighed ; they are again replaced and the test continued for half an hour. If the weight of the tube is constant the test is finished, but if there is any great increase in weight between the first and second weighing, they must again be replaced for another half hour. Deduct the weight of the tubes from their original weight, and this gives the weight of moisture in the coal taken. From this

the percentage is easily calculated. The third tube must not increase in weight at all, if so, it is doubtful if all the moisture has been absorbed.

The tubes on inlet likewise must remain constant in weight to be sure that no moisture is going into the coal.

If the tube with the coal in it is now weighed it will be found to have lost more in weight than the calcium chloride tubes have gained, showing that any method of drying

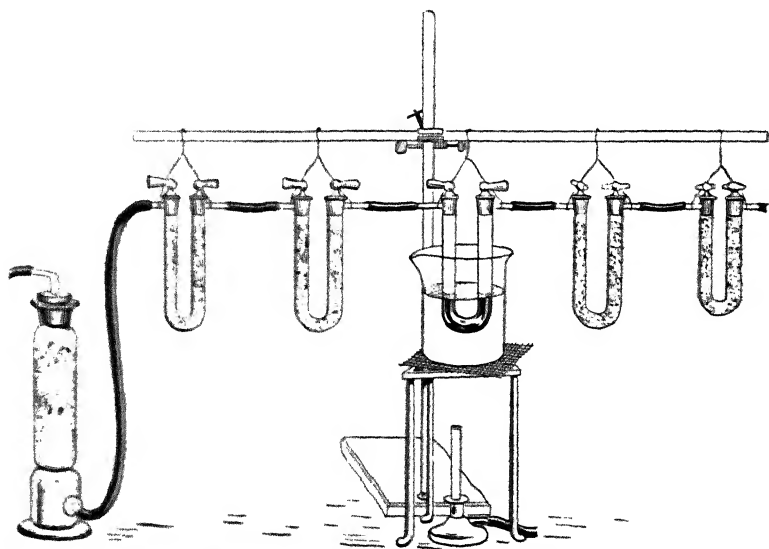


FIG. 2.—APPARATUS USED FOR ESTIMATION OF MOISTURE IN COAL.

coal and weighing the decrease in weight of the coal is obviously inaccurate.

For moisture in coke this is an excellent apparatus, but coke may be safely dried in the water oven. Without any error occurring, the loss of weight in coke would be solely due to moisture, as coke has already been subjected to a far higher degree of heat than it would obtain in the water bath.



Another excellent method is to dry the coal in a desiccator over sulphuric acid until the weight is constant. This method also only gives the free moisture. (Fig. 3.)

(2.) **Sulphur** in coal or coke is best determined by one of the following two methods. There are many and various methods, but these are undoubtedly the best.

(a.) *Nakamura*.—A small but average sample of the coal is ground in an agate until the whole will pass through muslin or a very fine mesh sieve. Most errors are caused through not grinding fine enough.

One gram is taken and mixed with about five times its weight of the following mixture:—13 parts of anhydrous potassium carbonate and 10 parts of anhydrous soda carbonate; this is well mixed with the coal and put into a large platinum crucible, and some of the mixture (without coal) is sprinkled on the top of the crucible. The crucible is then covered and the whole gently heated by the aid of a spirit lamp for two hours, and no smoke must escape from the crucible. The heat is gradually increased, and the contents should gradually fade to a greyish colour. The heat is then increased to redness for from one to one and a half hour, and the whole mass is then extracted with water, filtered, and bromine water added to oxidise sulphides to sulphates; boil to expel excess of bromine, acidulate with hydrochloric acid, and precipitate the sulphur by addition of barium chloride in excess as barium sulphate. Dry, burn off in a platinum crucible, and the weight of barium sulphate found  $\times 0.1373 \times 100$  gives the percentage of sulphur in the sample. The process depends upon the slow but perfect oxidation of the whole of the organic matter of the coal, and if any fumes are seen coming from

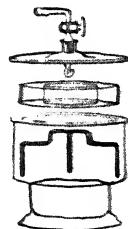


FIG. 3. — DESICCATOR USED FOR ESTIMATION OF MOISTURE IN COAL.

the crucible the test is worthless, as it shows imperfect oxidation.

(b.) *Modification of Eschka's Process.*—One gram of finely powdered coal (as before) is thoroughly mixed with 1 gram of light porous magnesium oxide and 0.5 gram of anhydrous sodium carbonate in a platinum crucible.

The crucible is very gradually heated by means of a spirit lamp, stirring the mass constantly until strong glowing has ceased. The temperature is then gradually increased until in about fifteen minutes the bottom of the crucible is of a dull red heat. After all the carbon has burnt away the mass is transferred to a beaker and dissolved in water. The liquid is then oxidised with bromine water; filter and wash filtrate, acidulate filtrate with hydrochloric acid, add barium chloride, and proceed as before.

These two methods are both suitable for the sulphur in the coke.

The difference between the sulphur in coal and the sulphur in the coke is the volatile sulphur, and special notice should be drawn to this in making the report on a sample of coal for gas making, as it necessitates extra expense in purification.

(3.) **Phosphorus.**—Ten grams of coal or coke are ignited in a platinum basin until only the ash is left; this is then fused and digested in a covered beaker with 20 c.c. of brominised hydrochloric acid for an hour at nearly boiling temperature. The bulk of the acid is then removed by evaporation, and the solution is diluted with 30 c.c. of water. The whole is then filtered, and the residue washed with distilled water, 15 c.c. of strong ammonia are added, and this is neutralised by nitric acid from a burette. When re-solution takes place, diluted ammonia is added drop by drop until a faint cloudiness or opalescence appears; this is then re-dissolved by addition of a few drops of nitric acid.

The liquid is kept near the boiling point during this operation.

To the faintly acid solution 3 c.c. of strong nitric acid are added, and then 5 c.c. of a 10 per cent. solution of ammonium molybdate is quickly added, the solution being briskly stirred.

After a second or two the yellow ammonium phosphomolybdate settles in a granular form. This is then nearly

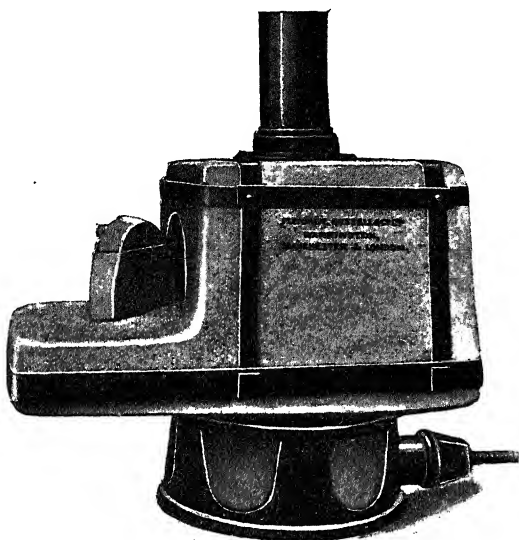


FIG. 4.—MUFFLE FURNACE FOR ESTIMATION OF ASH IN COKE.

boiled for five minutes. The precipitate is then filtered off on a tared filter paper, washed with a weak solution of nitric acid, dried on the water bath and weighed.

The weight of precipitate  $\times .163$  = per cent. of phosphorus (Stock method).

**Ash.**—Five grams of finely powdered coal or coke are weighed in a platinum basin and incinerated in a muffle furnace (Fig. 4) until constant in weight. This usually occupies

three to four hours. At the end of the third hour, place crucible in desiccator to cool, and weigh, then replace in muffle for one hour; cool and weigh again. The weight should be constant, if not, repeat operation until weight is constant.

**Volatile Matter and Fixed Carbon.**—It is sometimes useful to know these on a small scale. The method is :— 2 grams of coal are ignited in a platinum crucible, with

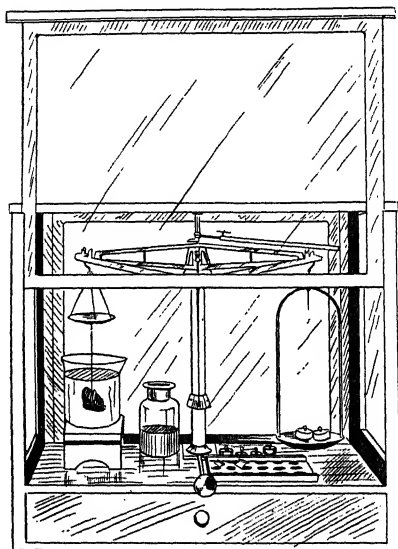


FIG. 5.—APPARATUS USED FOR ESTIMATION OF SPECIFIC GRAVITY OF COAL.

close fitting lid; heat gently for the first few minutes, then over a blast, taking care that the flame does not entirely envelop all the crucible, until all combustible gases are driven off; cool and weigh. It is necessary to always have the gas at the same height in every test, and three determinations must be made on the same sample and average taken. At the best the results are only approximate.

**Specific Gravity.**—The specific gravity of coal is ascertained on the following principle: that when a body is weighed, suspended in liquid, its weight is diminished by the weight of the liquid displaced. Therefore if the body is weighed first in air, and again when immersed in water, the difference between the two weights is the weight of water displaced by that body, *i.e.*, the volume of water

which is equal to the volume of the body. (Fig. 5.) Three or four pieces of the sample of coal (representative of the sample) are selected, about the size of a walnut. These are brushed free from dust, and one piece at a time is weighed first in air and then in water. The coal is suspended from the arm of the balance (of which one pan is removed and a special gravity pan used) by a silken thread. The weight of this in air is first taken, and a beaker full of water is placed under the coal and is nearly filled with water at 60 degrees Fahr., and the coal (which reaches to about the centre of the beaker) is then suspended in the water. It is necessary to remove all air bubbles, and the coal is moved up and down until such are all liberated. The coal is then weighed.

The specific gravity is then calculated as follows:—

Weight of coal in air	-	-	260.0 grams
Weight of coal in water	-	-	59.8 „
<hr/>			
Loss of weight in water	-	-	200.2 „
$\frac{260.0}{200.2} = 1.298^{\circ} \text{ specific gravity.}$			

**Nitrogen.**—The nitrogen in coal is usually determined by Kjeldahl's method. This depends upon the fact that many nitrogenous organic compounds, when heated with strong sulphuric acid, have their nitrogen converted into ammonia, which at once unites with the sulphuric acid, forming ammonium sulphate. The amount of ammonia in the ammonium sulphate is then determined by distilling with caustic potash into a standard sulphuric acid solution, and the amount of  $\text{NH}_3$  estimated; from this the nitrogen is calculated.

**Arsenic.**—It is sometimes of very great importance to know the amount of arsenic in the coke that is being supplied for certain technical purposes. The standard and official method adopted by the Government laboratory,

is known as Thorpe's method (*Journal Chem. Soc.*, 1903, p. 969).

By a suitable method of testing, the minutest trace of arsenic can be easily determined to amounts below one-thousandth of a grain per lb.

The method adopted by Thorpe is fairly rapid in execution and distinguishes the amount of arsenic which is volatile, and that which is fixed and is left in the ash.

A piece of hard glass tube, about 60 cm. long, is drawn out, and the drawn-out portion is bent into the shape of an adapter, as shown in Fig. 6, A. Ten grams of the finely powdered sample of coke is then introduced into the tube, so that it occupies the centre of the tube, leaving empty a certain space. A convenient method of introducing the fuel is to evenly distribute it on a piece of glazed cardboard (to prevent the substance adhering to it), which can be inserted in the tube and inverted, the coke distributed as desired, the cardboard being withdrawn.

The drawn-out portion is then connected, as shown, with the absorption apparatus containing dilute sulphuric acid. A convenient form of apparatus being a modification of De Koninck absorption bulbs, so that the products of combustion are offered a good wetted surface. The hard glass tube is placed in an ordinary combustion furnace and connected with an oxygen gasholder.

The burners beneath the empty portion of the tube are first lighted, and a rapid current of oxygen kept continually passing through the apparatus. The powdered fuel is then heated nearest the inlet of the oxygen, and as soon as the combustion once starts, very little heat will be required, and the coke burns away without the formation of sooty or tarry products.

The whole operation is under perfect control, and occupies from two to three hours, depending upon the nature of the coke.

The ash is left in a loose, pulverised form. The arsenic in the fuel is then partly in the ash and partly in the liquid

or the absorption apparatus, and partly in the end of the combustion tubing.

1. To determine the amount of the arsenic retained in the ash:—

The ash is shaken into a small Kjeldahl's flask of about 100 c.c. capacity, which is then attached (preferably by means of ground glass joints), as shown in Fig. 6, B, to a small condenser, connected to a small flask containing about 10 c.c. of hydrochloric acid (sp. gr. 1.1). Into the flask containing the ash 25 c.c. of hydrochloric acid containing 0.25 c.c. of strong bromine are added through the funnel D.

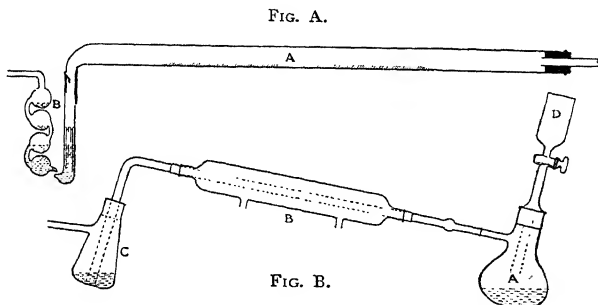


FIG. 6.—APPARATUS FOR ESTIMATING ARSENIC IN FUEL.

FIG. A.—A, Hard glass tube; B, Absorption tube.

FIG. B.—A, Distillation flask; B, Condenser; C, Flask for receiving distillate; D, Funnel.

The glass is then heated and the liquid maintained just on the boil for two hours. After it has cooled, add about a gram of potassium metabisulphite, and the liquid is again heated until all the free bromine disappears.

The solution is then filtered free from the insoluble or suspended silica, and the filtrate is washed with the acid contents of the other flask. It is not absolutely necessary to remove the silica, but it facilitates the working and prevents irregular boiling. The filtered solution is returned to the distilling flask and connected to the condenser, and is boiled to expel the sulphurous acid.

1114

665

N07

The condenser is then reversed and the liquid distilled into the small flask attached to the other end.

The distillation is continued until the residue in the distilling flask becomes syrupy, when 10 c.c. more of hydrochloric acid are added to the residue, and the distillation is again carried on.

The total distillate is made up to 100 c.c., and an aliquot part of this is transferred to a small porcelain dish, 5 c.c. of pure nitric acid and 2 c.c. of pure concentrated sulphuric acid are added, and the solution is evaporated until fumes of sulphuric acid are freely evolved.

The dish is cooled and diluted with 20 c.c. of water and transferred to a small flask.

Half a gram of potassium metabisulphite is added, and the solution boiled until free from sulphurous acid.

When cool this solution is ready for the determination of the fixed arsenic by means of Marsh's apparatus.

2. The amount of volatile arsenic in the combustion of the fuel is carried out as follows :—

The acid in the absorption tube is poured into a small beaker, and the absorption tube rinsed with a small quantity of water.

The end of the hard glass tube is then well washed by repeatedly sucking the liquid up from the small beaker into it.

Finally the glass tube is washed out with a little more acid, and the total washings made up to about 50 c.c. ; of this, 25 c.c. are taken and used directly for the estimation of the arsenic. The estimation of the arsenic in both solutions, or the fixed and the volatile arsenic (which are done separately), may be made by means of Marsh's apparatus, and in this case it is unnecessary to remove the hydrochloric acid by evaporation with nitric and sulphuric acid.

The nascent hydrogen required for Marsh's process (which is unnecessary to describe in detail, and can be found in any good text-book on chemical analysis) is conveniently prepared by the action of dilute hydrochloric acid on zinc,



mixed with clippings of pure electrotype copper in quantity to give a steady and fairly rapid stream of the gas.

The amount of arsenic obtained is estimated by comparison with arsenic deposits obtained from the use of arsenious oxide on the same apparatus.

It is hardly necessary to impress the necessity of carrying out blank experiments on the chemicals and reagents to ascertain that they are absolutely free from arsenic.

Thorpe has proposed and used an electrolytic method in the place of Marsh's apparatus. Briefly this apparatus consists of a circular glass vessel, provided with a ground glass stopper and connections, and carries a drying tube, filled with calcium chloride. This with a porous vessel forms the inner cell for the cathode where the hydrogen and hydrogen arsenide are produced on passing the electric current.

The vessel is opened at the top, and has, passing through the ground glass stopper, a tap funnel, the stem of which reaches to a point just below the neck of the vessel. From the stopper a bent glass tube, bulb shaped, passes off and is connected by means of a ground glass joint with the drying tube. A stout platinum wire, fused through the glass cap, establishes connection between the current generator outside and the electrode within the vessel.

A small Bunsen circular burner is used with this apparatus.

The standard deposits are made from definite strength solution of pure resublimed arsenious oxide, which is ground to a powder and dried at 100 degrees Cent.; 0.1 gram is accurately weighed and transferred to a litre flask, but washing it down into the flask with 1 to 2 c.c. of pure hydrochloric acid. The liquid must not be heated, and when the solution is complete, it is made up to a litre with distilled water. Each c.c. of this solution contains 0.0001 gram of arsenious oxide.

100 c.c. of this solution are now accurately measured

and diluted to 1,000 c.c. by distilled water. Each c.c. of this solution contains 0.00001 gram of arsenious oxide.

5 c.c. of sulphuric acid are diluted with 20 c.c. of water, 0.5 gram of potassium metabisulphite is added, and the solution is boiled to expel sulphurous acid. When cold the solution is tested for its freedom from arsenic.

Similar quantities of sulphuric acid are now taken, and to them in turn are added varying quantities of the standard arsenic solutions.

These are now tested for arsenic deposits, which are the standard deposits, and are tabulated and kept for reference.

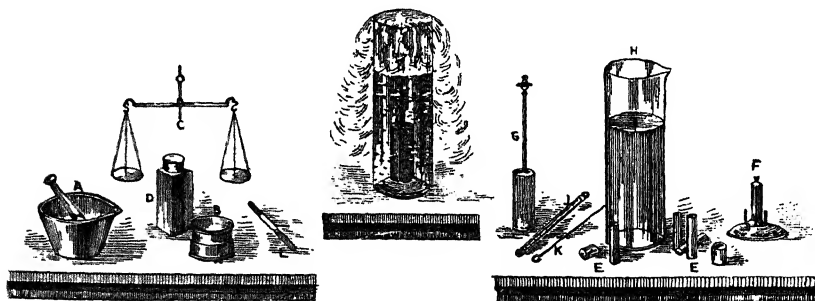


FIG. 7.—LEWIS THOMPSON'S FUEL CALORIMETER.

A, Pestle and Mortar; B, Sieve for sifting coal; C, Rough scales; D, Box containing combustion mixture; E, Cylinders for holding coal and combustion mixture; F, Stand with clips for holding cylinders; G, Cover to go over F; H, Water cylinder; J, Thermometer; K, Wire for cleaning G after combustion; L, Spatula.

The solutions obtained from the fuel to be tested are next tried for their amount of arsenic, and their deposits compared with the standard ones.

**The Calorific Value of Coke.**—There are many apparatus for the estimation of the calorific value of the coke, amongst the best known being the Lewis Thompson's calorimeter, Mahler bomb calorimeter, and various modifications of these.

The Lewis Thompson calorimeter consists of a cylindri-

cal copper furnace, a spring clutch base to carry a furnace, copper combustion cylinder or furnace fitting the spring of the base, a delicate thermometer, clearing wire, and graduated glass cylinder, and the oxygen mixture.

A representative sample is powdered so that it will pass through the sieve, and a smaller quantity is then ground to a very fine powder in an agate mortar. Two grams of this are thoroughly mixed with about ten times their weight of the oxygen mixture, which must also be of a fine powder and dry. The whole of the mixture is then placed in one of the copper furnaces and compressed down a little at a time.

The furnace is then placed in the socket of the brass plate. Place half an inch of fuse on the top, add a little oxygen mixture loosely around fuse. The measure is then filled with water to the desired mark, which holds 1,934 grams of water, and its exact temperature noted. If left to stand in the round some time it will be the same temperature as the room. Not more than 2 degrees is the difference between the air of the room and the water. Close stop-cock in combustion cylinder, light fuse, and quickly place the cylinder over brass base and furnace, and at once submerge the whole into the glass cylinder of water.

Combustion will have ceased as soon as bubbles stop rising from bottom of cylinder, usually in about a minute, open stop-cock, and move cylinder up and down in the water so as to equalise temperature. If the condenser does not vent when tap is opened, clear passage with wire.

The thermometer is in the water during the whole of the operation.

The difference in the temperature of water before and after + 10 per cent. of the difference (for the heat absorbed by the metal cylinder). Multiply total difference by 1.8 equals lbs. of water evaporated per lb. of fuel.

To find calorific value multiply total difference in temperature by 956 = calories per lb. of fuel.

$$\begin{array}{r}
 \text{Increase} = 5.6^\circ \text{ Cent.} \\
 10 \text{ per cent.} = .5 \\
 \hline
 6.1
 \end{array}$$

$$\begin{array}{r}
 6.1 \times 1.8 = 10.98 \text{ per lb.} \\
 6.1 \times 956 = 5,831.6 \text{ calories.} \\
 5,831.6 \times 1.8 = 10,596.88 \text{ B.T.U.} \\
 \text{per lb.}
 \end{array}$$

One of the best modifications of Mahler bomb is that known as the Mahler-Donkin bomb calorimeter.

The bomb in which combustion takes place is made of special metal, which resists corrosion and has a very high tensile strength. It is plated inside to withstand corrosion by the acid generated during combustion. It is fitted with a cover which fastens down. This cover has a screw valve attached to it to regulate the introduction of oxygen from a Brin's cylinder. The fuel is placed in a platinum crucible inside the bomb.

The electrodes are connected by a fine wire which serves to ignite the fuel, and are connected to a battery for this purpose.

One of the electrodes is insulated by a porcelain collar where it passes through the cover of the bomb.

The bomb is placed inside a vessel containing a known weight of water, which is stirred by an arrangement of paddles to ensure a uniform temperature. This vessel is in the interior of an annular vessel, which acts as a water jacket. There is also an air space between the inner vessel and the water jacket, which serves as a heat insulator, while the outer water jacket prevents heat reaching the calorimeter from external sources.

A thermometer is provided for taking the temperature necessary.

A certain amount of the fuel to be tested is ground to a very fine powder, and 1 gram is weighed out and placed in the platinum crucible.

The electrodes are now connected by means of platinum wire about 0.1 mgr. in diameter. The loop of the wire between electrodes should rest on the fuel in the crucible.

The bomb cover is now screwed down on a joint of fine lead wire.

The cylinder of compressed oxygen is now connected to the lower branch provided on the screw valve.

Connect up pressure gauge, open the valve on bomb cover, and then slightly open the valve of oxygen cylinder. Gently open the regulating valve and admit oxygen to bomb very slowly, and allow pressure to increase gradually by degrees. When the pressure is 25 atmospheres per

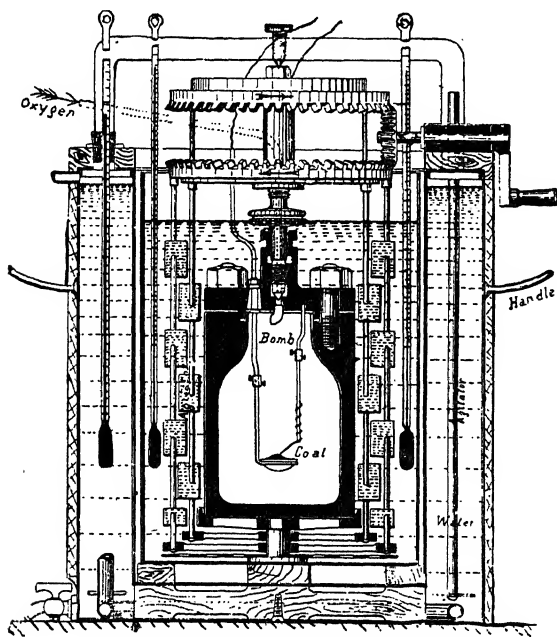


FIG. 8.—MAHLER-DONKIN BOMB CALORIMETER.

square inch, the supply of oxygen is cut off. This quantity is ample for the combustion of 1 gram of fuel.

Close regulating valve and then screw down the valve on bomb cover.

The oxygen cylinder and tubing may now be disconnected.

Weigh out the required amount of water, which is usually

from 2,000 to 2,500 grams, and pour into the calorimetric vessel. Then place bomb and stirring gear in position inside the vessel. The water jacket must be kept filled with water, and its temperature must be steady at that of the atmosphere. Well agitate water and read thermometer.

The fuel is now ignited by connecting one wire from battery to the insulated terminals, and with the other make contact with any part of the bomb cover. Combustion immediately starts.

The temperature is taken every half minute until thermometer begins to fall.

The maximum temperature is carefully noted.

The stirring apparatus must be kept at work during the whole of the period.

The formula for calculating the results, with no correction, and these results are sufficiently accurate for all practical purposes :—

If  $W$  = weight of fuel tested in pounds.

$H$  = calorific value of fuel in B.T.U. per pound.

$W_1$  = weight of water in calorimetric vessel in pounds.

$W_2$  = water equivalent of calorimeter in pounds.

$t_0$  = temp. Fahr. of water in calorimeter before combustion.

$t_1$  = maximum temp. Fahr. of water in calorimeter after combustion.

Then  $(W_1 + W_2) \times (t_1 - t_0)$  = heat received by water and instrument.

$WH$  = heat evolved by combustion of fuel sample.

Hence  $H = \frac{(W_1 + W_2)}{W} \times (t_1 - t_0)$  in B.T.U. per pound.

## CHAPTER III.

### *FURNACES—TESTING AND REGULATION.*

It is not proposed to deal in any way with the construction of retort furnaces and settings, but only to give the best methods of regulating these settings from a practical scientific point of view. If proper attention is paid to this important point on a gasworks, a considerable saving can be made in the fuel used in the furnace, giving a correspondingly larger quantity of coke for sale. There are various types of settings in use at the present time. The following is a brief description of their characteristics:—

1. Simple direct coke-fired setting, with open grate.
2. The generator type of setting where solid fuel is gasified, but no means for heating primary or secondary air.
3. The regenerator or recuperative setting in which the fuel is gasified as in generator, and in addition the waste gases are utilised to heat the supply of secondary air, and in some cases the primary air also, thus returning to the setting heat which would otherwise be lost. The principle of the two latter types of gaseous firing is the conversion of the carbon in the coke into carbon monoxide at first, and then by the proper admission of secondary air to convert this  $\text{CO}$  to  $\text{CO}_2$  in the combustion chamber.

In the old type of furnace (open grates) the coke is burnt to  $\text{CO}_2$  direct in the furnace, and it is necessary to have the retorts as near to this source as possible.

The uncontrolled inrush of cold air, the smallness of the furnace necessitating the frequent opening of the door for

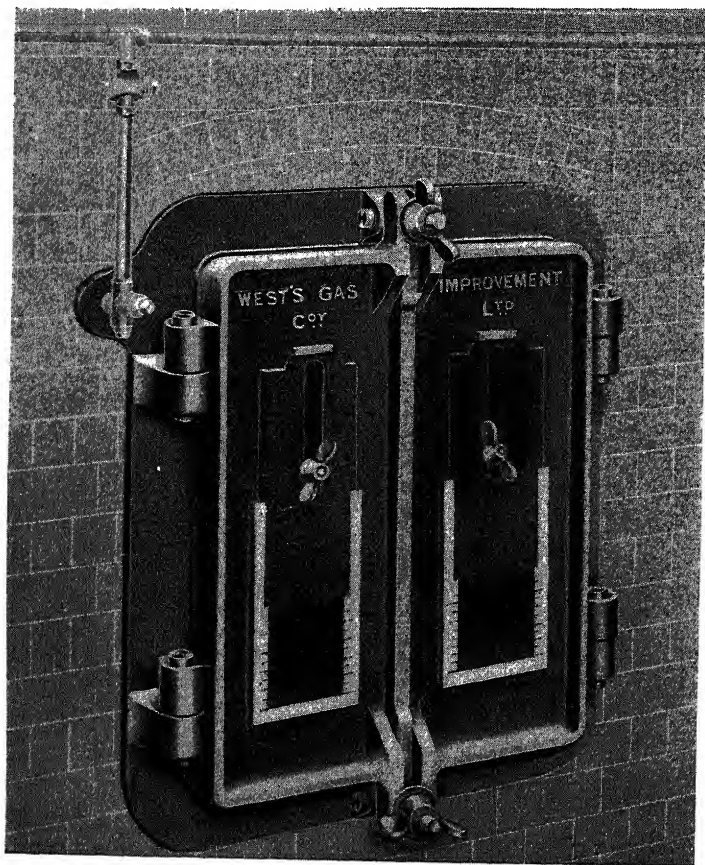


FIG. 9.—TIGHT CLINKERING DOOR, SHOWING PRIMARY AIR PORTS.

recharging and clinkering, the use of cold coke, all combine to cause great waste of fuel and labour.

From the chemist's point of view very little can be done with this type of setting.



In modern works, direct fired settings have been entirely superseded by the system of gaseous firing, the advantages of which are readily understood.

The large furnaces comparatively seldom require clinkering and feeding; the use of hot coke and the admission of just sufficient air for the necessary combustion result in large economies in fuel composition and wear and tear. A form of gaseous firing is the generator, and the more advanced type is the regenerator.

The settings have an absolutely tight clinkering door through which are cut ports to admit the primary air, sufficient to convert the fuel into carbon monoxide.

The combustible gases pass from the generator into the combustion chamber, and it is here that the secondary air is admitted, and the final combustion takes place.

The high temperature flame thus formed circulates round the retorts, heating them evenly, and without the cutting action so noticeable in the direct fired setting.

If this secondary air is heated by the waste gases, further economies are then obtained, and where this is done the setting is termed the regenerator type. In these settings the waste gases are conducted through passages on their way to the exit or main flue. These passages run along the side of the secondary air flue, so that the secondary air absorbs a certain amount of the heat from the waste gases, and instead of entering the combustion chamber cold, it has now attained somewhere about the temperature the waste gases are leaving. The secondary air travels in a directly opposite direction to the exit gases, as shown in the illustration.

The saving in fuel consumption with this latter type of setting is considerable.

Another advantage of gaseous fired settings is that the length of flame in the combustion chamber can be controlled, and a large number of retorts grouped in the oven and evenly heated. After this brief glance at the various types of settings comes what was originally intended to

show, viz., the testing and regulation of these settings from a chemist's point of view. The direct fired setting has been previously dealt with and needs no further discussion. It is not proposed to go fully into the benefits, &c., of various furnaces, but it will be as well to point out here that the

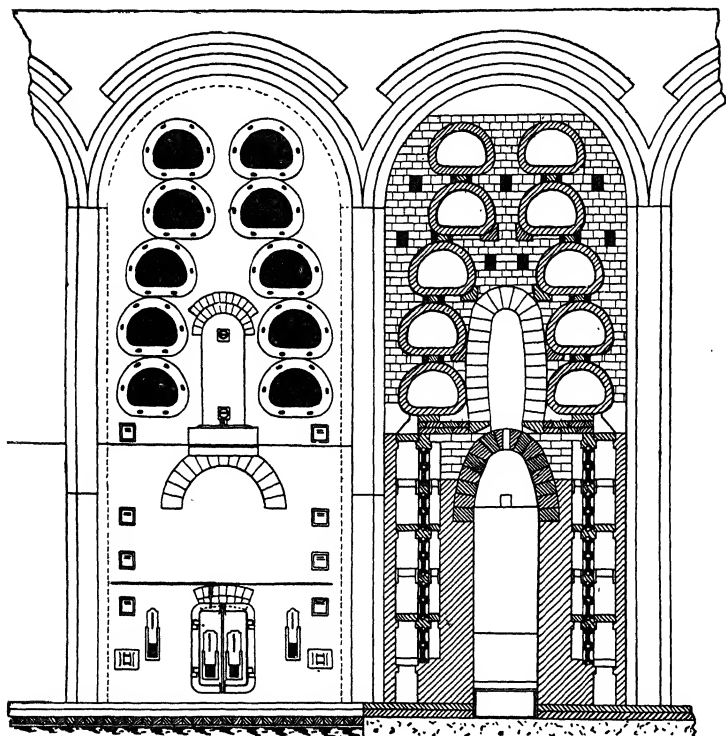


FIG. 10.—SECTIONAL ELEVATION OF RETORT SETTING.

highest temperature attainable, theoretically, is about 5,300 degrees Fahr., from which one has to deduct about 1,200 degrees Fahr. (being approximately the temperature of gases in the exit flue), leaving 4,100 degrees Fahr. used by the setting, showing a loss of about 26 per cent.

In the generator type of setting we have a good depth of fuel, of between 3 ft. 6 in. to 5 ft. The furnace door is made air-tight, as far as is practically possible, and is only opened for clinkering and pricking up purposes. The

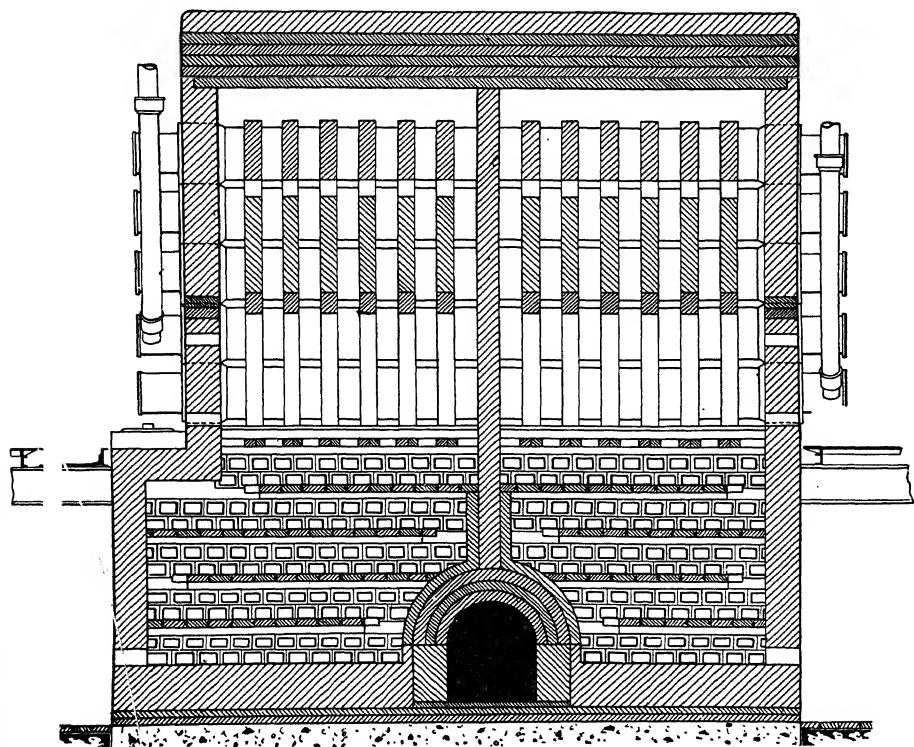


FIG. 11.—SECTIONAL ELEVATION OF RETORT SETTING, SHOWING FLUES AND PASSAGES

furnace is fed through the sleeve in the top of the producer with hot coke direct from the retorts.

On either side of the generator walls are placed the secondary air flues. The function of these flues is to abstract as much heat as possible from the waste gases. The waste gas flues are under the bottom retorts of the

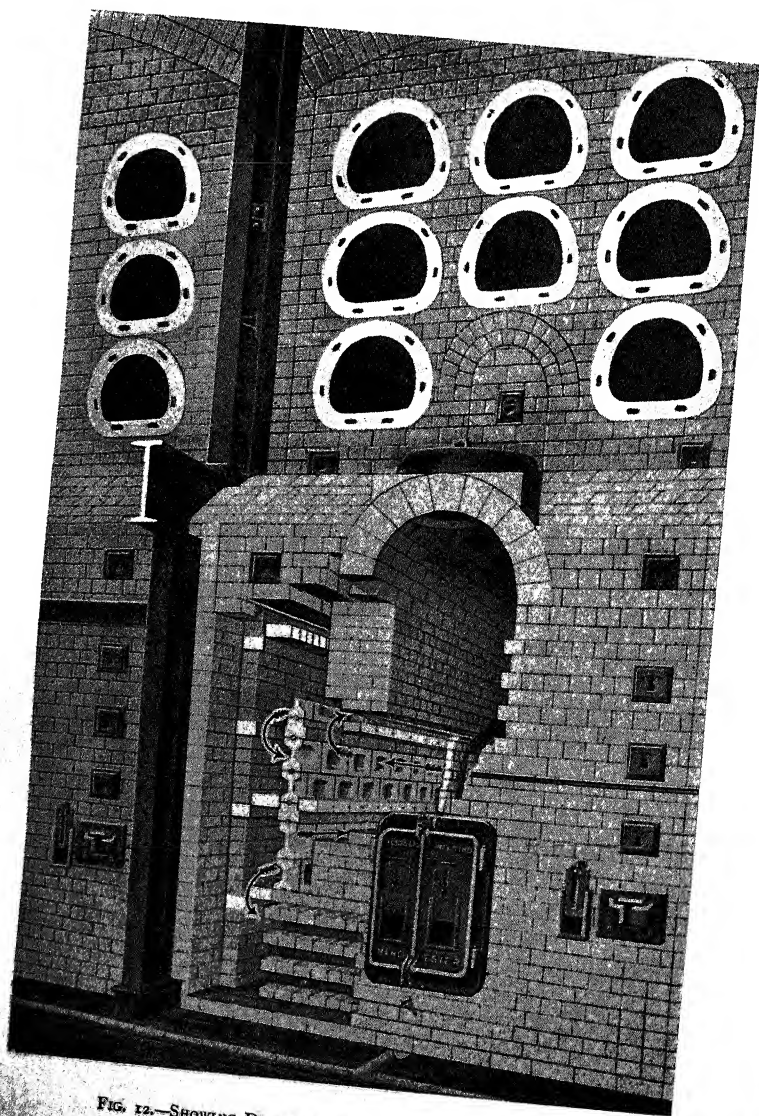


FIG. 12.—SHOWING DIVIDING WALLS FOR WASTE GAS FLUE.

setting, and are as near as possible to the secondary air flues. These flues go down the side of the setting and they exit at the bottom into the main flue.

Every unit of heat abstracted from the waste gases and returned to the combustion chamber by increased heat in the secondary air is so much gain. To keep down the temperature in the producer steam is usually supplied by means of a continuous stream of water, which drops or runs on to the fire-bars, which are usually V or U shaped.

The action of the steam formed is to prevent the formation of clinker at the fire-bars by cooling the bars and fuel immediately above them, raising the zone of highest temperature some little distance above the bars, and rendering the clinker friable and easily removed without damage to the bars, and less labour.

In the regenerator type of setting we have a greater depth of incandescent fuel, and consequently a more perfect action in the producer. By their slow travel alongside of the secondary air flues the waste gases become reduced in temperature, and should leave the setting at about 500 degrees Fahr.

The dividing walls should be as thin as possible, whilst at the same time possessing reliable joints. A good example is shown in the illustration. To prevent short circuiting as much as possible, the flues and passages should be periodically examined and washed out, where practicable, with a good fire-proof cement.

Should by-passing take place it will probably result in insufficient secondary air reaching the combustion chamber, and the burning of the producer gas in the waste gas flues, instead of in the combustion chamber, causing a hot main flue and bad heats in the settings.

Excessive heat in the main flue, *i.e.*, where it is not wanted, means, of course, a waste of fuel.

Every effort should be made, whilst keeping sufficiently high heats on the setting, to prevent the heating flame from reaching the main flue. This nicety of adjustment is

arrived at principally by the frequent analysis of the gases at various points of the setting, carried out as hereafter described.

Faulty heats can rarely be improved by increasing the damper; they should rather be remedied by proper adjustment of the primary and secondary air, and attention to the clinkering and pricking up of the fire.

**Analysis of Furnace Gases and Waste Gas from a Regenerator Setting.**—The sampling of a gas is no easy task, and one must take certain precautions to ascertain that the sample is a true and representative one, and feel confident that there is nothing amiss in the method employed.

The furnace gases being of a fair temperature (verging on 2,000 degrees Fahr.) iron tubes will not do. The most suitable is undoubtedly a platinum one, but as this is generally out of the question, the next best is a porcelain tube. A porcelain tube of about 4 ft. long, with a diameter of about 1 in. This is then wrapped round with asbestos, or better still is placed inside a brass or iron tube and packed with asbestos. It is necessary to have a fire-brick with a hole in it to take the tube. The joint is made tight by fire clay or "pug."

In taking a sample of gas from the producer, it is necessary to have a special iron door which fits on to the "sleeve" or charging aperture in the producer. The tube is put through this, and the whole made tight. The tube should then be in the top of the "horse-shoe" above the coke.

The aspirator (preferably) of glass, which has a capacity of a gallon, is now connected on to the tube, and the water in the aspirator run out; the gas from the producer is then drawn into the space which is left free by the water, and after about three-quarters of the aspirator has run out, the aspirator is shut off, and the sample is tested. A similar proceeding is gone through with another aspirator and tube

on the exit gases at the *same time*, so that one has comparison in the results.

The condition of furnace is noted, such as when last cleaned, filled, pricked up, &c.

These samples are now tested, and undoubtedly one of the easiest apparatus for this is the "Orsat Muencke," or one of its many varieties. This apparatus gives very good results in non-technical hands, and any workman of average intelligence can be worked in to its use.

The apparatus (Fig. 13) is enclosed in a wooden case, with sliding shutters at each side, and is handy for transportation from place to place.

Its greatest advantage is that the measuring tube is jacketed with water which prevents changes of temperature affecting the gas volume.

The apparatus consists of a levelling bottle, the burette and four absorbing pipettes or bulbs, and the fourway connecting tubes.

The pipettes are filled about half-way with the required reagent, the first one filled with potassium hydrate ( $\text{KOH}$ ), the second one with an alkaline (potash) solution of pyrogalllic acid, and the third one with cuprous chloride; the fourth is not used for these analyses. To adjust the level of the reagents in the pipettes, which should be about midway between the top of the pipette and the rubber connection :—

This is attained by opening wide the cock upon the connector, the levelling bottle being on the table, and very gradually lowering the bottle until the reagent is brought to the required level; this operation is carried out for each

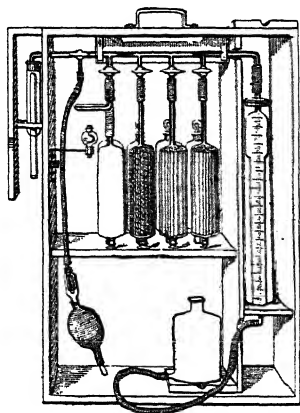


FIG. 13.—"ORSAT MUENCKE" APPARATUS.

pipette. The reagents being thus adjusted, the burette and connecting tube are completely filled with water by opening the cock and raising the levelling bottle. The apparatus is now ready to receive a sample of gas.

The aspirator is connected on to the inlet of the four-way piece (all the cocks on the pipettes being shut). The levelling bottle is now lower, and a sample of the gas is drawn into burette.

This is manipulated until the reading is exactly 100 c.c.; this is easily arrived at by lowering or raising the levelling bottle. The level of the water in this bottle must be at the same level as the water in the burette; shut inlet cock, and take the reading on burettes. The levelling bottle must be kept level with zero mark during reading, for if the bottle is raised the gas is compressed, and if the bottle is lowered it is expanded.

*Determination of Carbon Dioxide (CO<sub>2</sub>).*—The producer gas to be analysed is first passed into the first pipette, which contains potassium hydrate and will absorb the carbon dioxide, by opening the cock connecting this pipette with the main fourway piece and raising the levelling bottle, which drives the gas from the burette into this pipette, displacing the reagent in the front part of the pipettes, leaving bare the capillary tubes, which, being covered with the reagent, present a large surface to the gas. The reagent moves into the rear arm of the pipette. The levelling bottle is raised and lowered two or three times, forcing the gas in and out of the pipette, and the reagent brought to its original place on the stem of the pipette, cock shut, and burettes read. The difference between this and the first reading gives the percentage of CO<sub>2</sub>. To be absolutely certain that the CO<sub>2</sub> is all absorbed the operation is repeated. The reading must agree to 0.10 per cent., or else the operation must be repeated until such is the case. To ensure a fair amount of accuracy the burette ought to be allowed two minutes to drain, or otherwise the reading will be too low.



*Determination of Oxygen ( $O_2$ ).—*The residual gas after absorbing the  $CO_2$  is next passed into the second pipette, containing an alkaline solution of pyrogallic acid made by dissolving pyrogallic acid in potassium hydrate.

Proceed as before. The difference in reading gives the percentage of  $O_2$ .

*Determination of Carbon Monoxide (CO).—*The residual gas after absorbing the  $O_2$  is passed into the third burette containing cuprous chloride.

Proceed as before. The difference in reading gives the percentage of CO.

Now fill up burette with water, blowing the remainder of the gas away, and take a sample of the exit gases and analyse that in the same way.

The analyses one would expect from a good regenerative setting are :—

<i>Producer Gas.</i>		<i>Waste Gas.</i>	
$CO_2$ -	1 to 2 per cent.	$CO_2$ -	25 to 27 per cent.
$O_2$ -	nil.	$O_2$ -	0.40.
CO -	26.00.	CO -	nil.

**Interpretation of Results.**—It is waste of time analysing furnace and waste gases if the results are not properly understood. The theory of gaseous firing is to convert the carbon in the coke into carbon monoxide (CO), and afterwards, and in the right place, to burn this to carbon dioxide by proper admission of oxygen in the form of air.

The analysis of an ordinary sample of coke is as follows :—

Carbon	-	-	-	-	-	-	88.3
Hydrogen	-	-	-	-	-	-	0.2
Nitrogen	-	-	-	-	-	-	1.1
Water	-	-	-	-	-	-	3.5
Sulphur	-	-	-	-	-	-	0.4
Ash	-	-	-	-	-	-	6.5

---

100.0

The oxygen exists united with the hydrogen in the form of water.

The accepted calorific value of the various constituents are:—

Carbon	-	-	14,500	B.T.Units per lb. to carbon dioxide.
Carbon	-	-	4,303	" " " monoxide.
Carbon monoxide	-	-	4,370	" " " dioxide.
Hydrogen	-	-	52,000	" " " steam.
Sulphur	-	-	3,996	" " " sulphur dioxide.

It will be seen from the above that the richer the coke is in carbon better efficiency can be obtained in the setting or furnace.

When the furnace is charged with coke, the primary air issuing under the bars first of all combines with the carbon, and forms carbon dioxide:— $C + O_2 = CO_2$ .

On further progress through the bed of incandescent coke, it takes up more carbon and forms carbon monoxide:— $CO_2 + C = 2CO$ .

So it is easily seen that the less  $CO_2$  that one has in the producer gas the more carbon monoxide is available for combustion, and it is obvious the aim should be to keep the  $CO_2$  as low as possible in the producer.

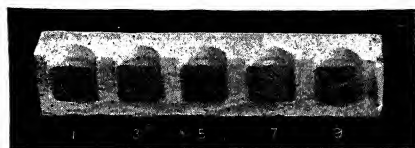
If this is high it shows one of two evils; either there is too much primary air (and consequent direct combustion will be noticeable by excessive heat in the producer), or this effect may be produced by insufficient depth of fuel or the chemical reaction to take place.

In the analysis of the waste gases, which must show that no carbon monoxide is going into the main flue, the heat is wanted in the combustion chamber and retorts, and it is necessary that sufficient secondary air be admitted to burn all the carbon monoxide to carbon dioxide in the proper place, and the analysis should show a slight excess of between 0.4 to 0.6 per cent. of oxygen in the waste gases. There will still be sufficient heat in the waste gases to give the desired draught.

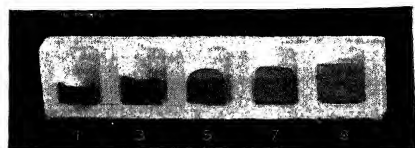
It is never advisable to control a setting by the use of the damper, but rather by the proper adjustment of the primary and secondary air inlets.

The damper should be set so that there is a slight pressure in producer, which can be observed through the sight plugs or the charging aperture or "sleeve." Sufficient has been said to show the importance of furnace gases analyses and the interpretation to be put on them.

It is often necessary to ascertain the heat in the setting and waste gas flues, as it is generally desired to work to as high a heat as the material will sustain.



Section Before Firing.



Section After Firing.

FIG. 14.—WATKINS' PATENT HEAT RECORDER.

There are numerous apparatus on the market for this purpose. One of the simplest is Watkins' patent heat recorder, and another of similar type is the Seger cone pyrometer. These are both on the same principle, viz., the use of various substances, the fusing points of which are known, and a series of these are grouped and marked so that it is easy to ascertain the temperature by noting which one fuses.

The heat recorder patented by Mr Henry Watkins of Burslem (Patent No. 6,288, 1900) consists of rectangular

blocks, each having five cylindrical recesses. Each recess has a number which denotes (in the table appended thereto) the temperature at which the test piece in the recess will fuse.

The cases are numbered consecutively, each case forming a series with a considerable range of temperature.

The test pieces or pellets are loose in case before fusion, and are wrapped in paper to prevent them falling out during transit, and the paper should not be removed; this will burn off.

These test pieces are composed of silicates, and are of definite chemical composition, and are tested before being sent out.

The duration of test in all cases is five minutes. The method of making a test is by using a bar of iron flattened at one end, on which the recorder is placed, and which is then placed in the flue or place which is desired to be tested.

In making a test, for example, suppose No. 11 pellet has fused, and No. 13 remains intact, the temperature on referring to the table will be between the two, viz., 1,634 degrees Fahr., or between that and the next (No. 13 = 1,742 degrees Fahr. To ascertain which series of the recorder should be used, the ranges of each are given, and it may be necessary to try the series below or above, as the colour is only approximate:—

A series	-	-	-	Faint red to bright.
B	"	-	-	Bright red to low orange.
C	"	-	-	Low orange to bright orange.
D	"	-	-	Bright orange to white.
E	"	-	-	Dazzling white.

Series and Numbers.	Temperature.		Series and Numbers.	Temperature.			
	Degrees Centigrade.	Degrees Fahrenheit.		Degrees Centigrade.	Degrees Fahrenheit.		
A.	1 -	590	1,094	C.	27 -	1,230	2,246
	3 -	650	1,202		29 -	1,270	2,318
	5 -	710	1,310	D.	31 -	1,310	2,390
	7 -	770	1,418		33 -	1,350	2,462
	9 -	830	1,526		35 -	1,390	2,534
B.	11 -	890	1,634	37 -	1,430	2,606	
	13 -	950	1,742	39 -	1,470	2,687	
	15 -	990	1,814	E.	41 -	1,510	2,750
	17 -	1,030	1,886		43 -	1,550	2,822
	19 -	1,070	1,958		45 -	1,590	2,894
C.	21 -	1,110	2,030		47 -	1,630	2,966
	23 -	1,150	2,102		49 -	1,670	3,038
	25 -	1,190	2,174				

Another method of easily determining the temperature of a retort or any part of the setting is by using Seger cones. Professor Dr H. Seger and E. Crammer, after numerous experiments, employed various earthen silicates, moulded into the form of tetrahedra or triangular pyramids. By varying the proportions of the ingredients in the mixtures forming these pyramids, it is possible to vary the temperature at which they melt. Every substance has a definite melting point, which is always the same provided the substance be pure. By adding to it another substance, we obtain a mixture which possesses a different melting point. By adding yet other substances and varying the proportions in which such substances are introduced, we can produce resultant mixtures which will always melt at definite temperatures.

These are the facts which have been relied upon, resulting in the production of the Seger cones.

Seger cones have melting points which range from 500 degrees Cent. to the melting point of platinum.

The following Table shows which cones are most adapted for use with various classes of goods :—

Porcelain colours and lustres, Nos. 022 to 010.

Goods manufactured from clays and containing a considerable amount of lime and iron oxide (e.g., stove tiles), Nos. 015 to 01.

Goods manufactured from clays poor in lime and iron, e.g., floor tiles, Nos. 1 to 10.

Stoneware with salt or loam glaze, Nos. 5 to 10.

White stoneware, &c. (hard burnt), Nos. 3 to 10.

White stoneware, &c. (mild burnt), Nos. 010 to 01.

Cement and porcelain, Nos. 10 to 20.

Refractory glazes, Nos. 20 to 25.

For determining the fire-resisting power of refractory earths, Nos. 26 to 36.

TABLE SHOWING MELTING POINTS OF SEGER CONES.

No. of Cones.	Melting Point. Degrees Cent.	Melting Point. Degrees Fahr.	No. of Cones.	Melting Point. Degrees Cent.	Melting Point. Degrees Fahr.	No. of Cones.	Melting Point. Degrees Cent.	Melting Point. Degrees Fahr.
022	590	1,094	02	1,110	2,030	19	1,510	2,750
021	620	1,148	01	1,130	2,066	20	1,530	2,786
020	650	1,202	1	1,150	2,102	21	1,550	2,822
019	680	1,256	2	1,170	2,138	22	1,570	2,858
018	710	1,310	3	1,190	2,174	23	1,590	2,894
017	740	1,364	4	1,210	2,210	24	1,610	2,930
016	773	1,419	5	1,230	2,246	25	1,630	2,966
015	800	1,472	6	1,250	2,282	26	1,650	3,002
014	830	1,526	7	1,270	2,318	27	1,670	3,038
013	860	1,580	8	1,290	2,354	28	1,690	3,074
012	890	1,634	9	1,310	2,390	29	1,710	3,110
011	920	1,688	10	1,330	2,426	30	1,730	3,146
010	950	1,742	11	1,350	2,462	31	1,750	3,182
09	970	1,778	12	1,370	2,498	32	1,770	3,218
08	990	1,814	13	1,390	2,534	33	1,790	3,254
07	1,010	1,850	14	1,410	2,570	34	1,810	3,290
06	1,030	1,886	15	1,430	2,606	35	1,830	3,326
05	1,050	1,922	16	1,450	2,642	36	1,850	3,362
04	1,070	1,958	17	1,470	2,678	37	1,870	3,398
03	1,090	1,994	18	1,490	2,714	38	1,890	3,434

In order to determine what cones are required when

desiring to regulate the burning of any setting or furnace, &c., it is necessary for the first few times to place a few cones in the setting, or wherever it is, and surround them with bricks (Fig. 15) or a muffle, or any arrangement that will keep them from being in direct contact with the flame, and then observe how they behave. If, for example, they exhibit the following appearance (Fig. 16), the conclusion might be drawn that the temperature agreed with the melting point of cone No. 7, because cone No. 6 is completely melted, whilst cone No. 7 has bent over so that its apex tends to approach the base, and cones Nos. 8 and 9 have remained erect and with their edges sharply defined.

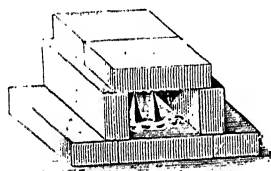


FIG. 15.—SEGER'S CONES IN USE.

Suppose it were found that on examining the goods that the fire was stronger than usual or heat too great, then

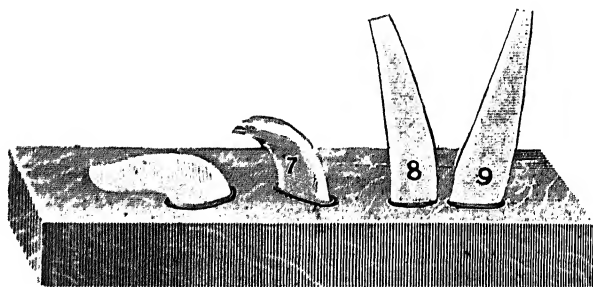


FIG. 16.—SEGER'S CONES AFTER FIRING.

after reducing the heat by various means another set of cones could be put in, for example, cones Nos. 4, 5, 6, and 7, and note what happened here. Supposing No. 6 was the one whose apex tends to touch the base, that would then be the temperature, viz., 1,250 degrees Cent. or 2,282 degrees Fahr. The other cone No. 7

could be left in and occasionally observed to see if heat increased.

The cones must always be placed in such a position that they are exposed to the full heat, and they do not encounter any live flame. The cones can be easily placed in the flues where they are protected from direct flame, and the heat of the secondary air easily ascertained, also exit gases, &c.

These cones can also be used to ascertain the heat in the producer, and can be lowered down into the producer through the feeding sleeve, the cones themselves being

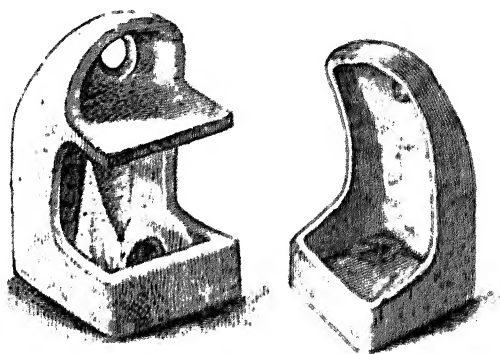


FIG. 17.—RECEPTACLES FOR SEGER'S CONES.

contained in a fire-clay receptacle which should preferably be covered, as, if the cones are touched in the heated state by dross or ashes, their melting point will be affected. The receptacles most commonly used are such as are shown in Fig. 17.

There are many electrical pyrometers, amongst the best being F  ry's radiation pyrometer, and Siemens' electrical pyrometer.

**The Siemens electrical pyrometer** is a platinum resistance thermometer, of which the essential element is a coil of platinum wire wound upon a cylinder of refrac-



tory material, and protected by a long closed tube. For measuring the resistance of the coil in order to ascertain its temperature, two types of apparatus are made.

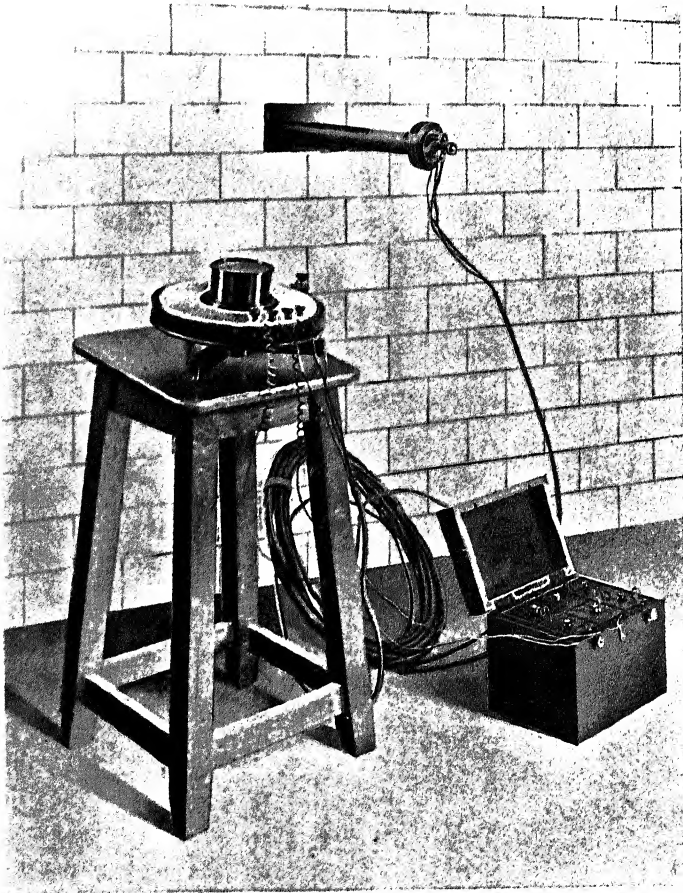


FIG. 18.—SIEMENS' ELECTRICAL PYROMETER IN USE.

The first comprises a differential galvanometer and a set of resistance coils, and gives readings in ohms, from

which the temperatures are ascertained by means of a special table supplied with the apparatus (Fig. 19).

The second is a combination of a small D'Arsenval

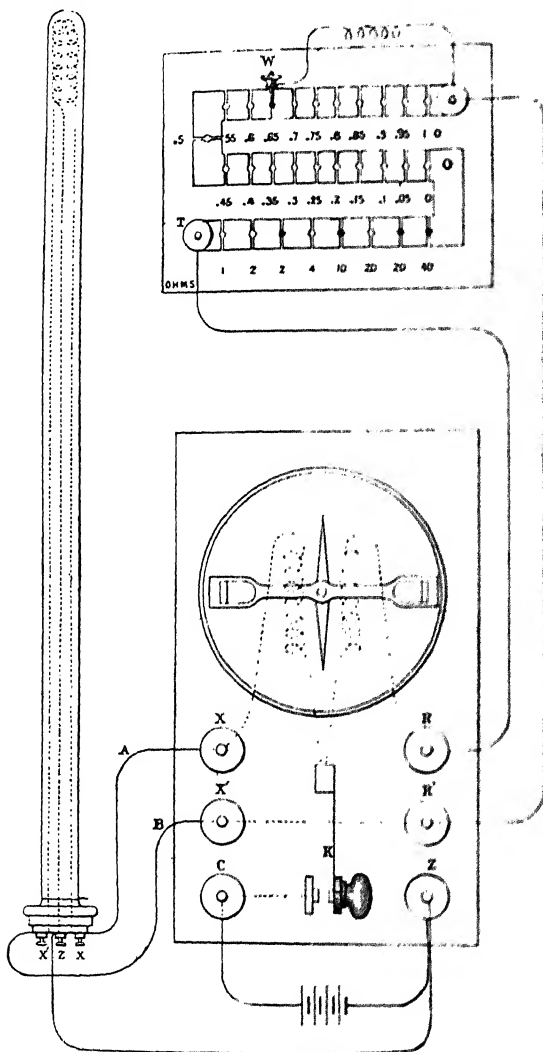


FIG. 19.—DIFFERENTIAL GALVANOMETER.

galvanometer and a Wheatstone bridge of circular form with sliding contact, and gives readings directly in Fahrenheit or Centigrade degrees as desired. Fig. 20.

### Diagram

showing Bridge connection

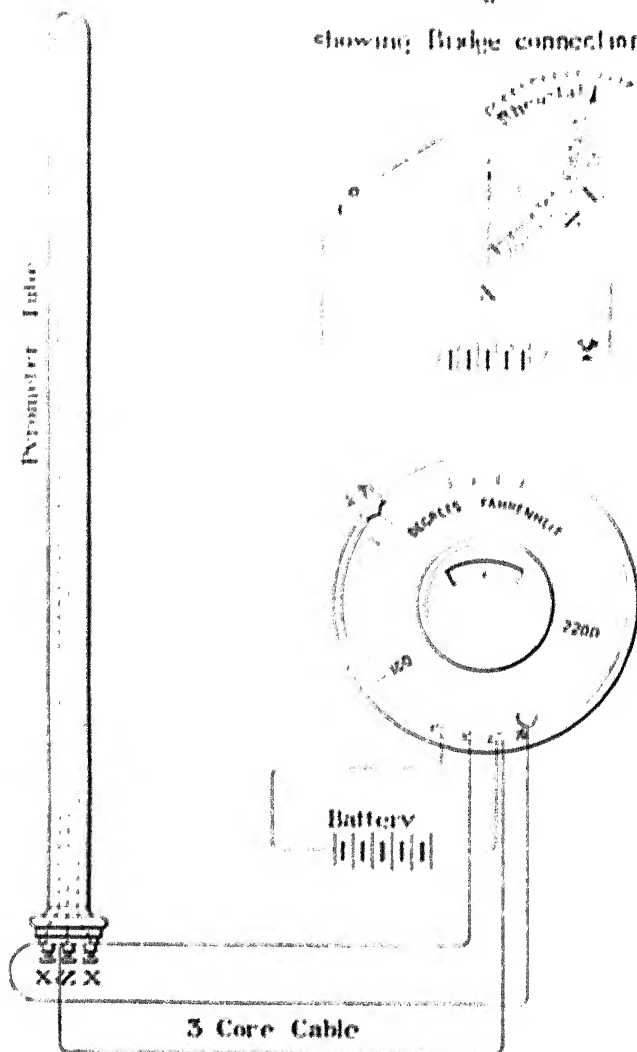


FIG. 20.—D'ARSONVAL GALVANOMETER

The pyrometer is most accurate; its reading can be depended upon to within a fraction of 1 per cent. It is also very simple in its action and management and its working, and may be safely entrusted to the care of an intelligent workman.

The method of connecting up the pyrometer with the differential galvanometer is shown in figure. Starting at the negative pole of the battery, a lead runs to the terminal *z* of the pyrometer tube, from which a wire extends to one end of the platinum resistance wire. At this point the circuit divides into two branches. One branch contains a wire running straight back to the pyrometer terminal *x'*, whence a lead *B* extends to the measuring apparatus, and is connected, through a resistance box and one coil of the differential galvanometer, with the positive pole of the battery.

The other branch includes the platinum resistance wire, a wire running to the pyrometer terminal *x*, and a lead *A* extending to the measuring apparatus, where it is connected through the second coil of the differential galvanometer with the positive pole of the cell. When the resistances of the two branches are equal, equal currents will flow through the coils of the galvanometer when the key *K* is depressed, and no reflection will be produced. The leads *A* and *B* from the pyrometer tube to the measuring apparatus are made of the same material and lie close to each other, so that their resistances are always equal, whatever may be their common temperature. The two wires within the pyrometer tube are also similar in all respects, so that their resistances always remain equal.

Thus it appears that the only independently variable resistances in the two branches of the circuit are the platinum spiral in one branch, and the resistance box in the other; the resistance unplugged in the box is thus equal to that of the platinum spiral when no deflection occurs in the galvanometer on depressing the key *K*. The

direct reading instrument comprises a Wheatstone's bridge and the necessary leads to the pyrometer tube.

Starting from the positive pole of the battery, a lead extends to the terminals *Z* of the pyrometer tube, whence a wire is connected to one end of the platinum resistance spiral. This junction forms one angle of the Wheatstone's bridge lozenge (see small diagram).

From it extends a wire running straight back to the pyrometer terminal *x<sub>p</sub>*, whence a lead extends to an arm which makes contact with a helical coil of wire arranged round the edge of a circular dial, thus forming one arm of the Wheatstone's bridge.

The conjugate arm of the bridge contains the platinum resistance wire, a wire running to the pyrometer terminal *X*, and a lead which extends back to the apparatus.

The two remaining arms of the bridge are made up of equal and constant resistances, the galvanometer being connected up as shown.

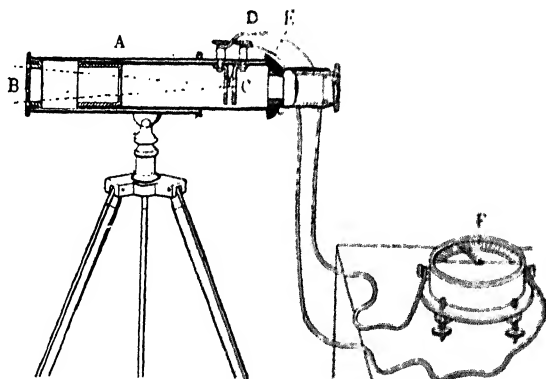
When no deflection is produced in the galvanometer on depressing the key, the two variable arms of the bridge must have equal resistances, since the resistances of the remaining arms are always equal; and since the two variable arms comprise equal leads, it follows that the resistance of that part of the helical coil thrown into circuit by the contact arm must be equal to the resistance of the platinum spiral in the pyrometer tube.

**The Féry Radiation Pyrometer.**—This instrument was invented by Professor Féry of the *École de Physique et de Chimie*, Paris. It is capable of taking very high temperatures, and consists of two adjustable lenses and a thermo-couple made of constantan (an alloy of 60 per cent. of copper and 40 per cent. of nickel) and copper wires.

In temperatures where the resistance thermometers cannot be supplied, with this pyrometer the height of the temperature does not matter, as no part is raised more than 80 degrees Cent. above the air temperature, and the pyro-

meter is not exposed to the action of the flue gases, and it is therefore a very valuable instrument in measuring high temperatures in the retort house.

The radiation emerging through a sight hole in the furnace is focussed upon the very sensitive thermo-couple, which is supported and protected from extraneous rays by screens. When the adjustment is correct, the image of the



Pyrometer for Laboratory Use.

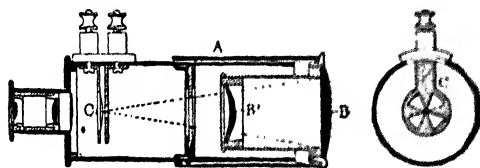


FIG. 21.—THE FÉRY RADIATION PYROMETER.

observation hole should completely cover the junction. The thermo-couple is connected to a Meylan-D'Arsonval galvanometer, divided into millivolts and degrees Centigrade for direct reading.

The horseshoe magnet has a single air space, and the movable coil of the galvanometer is balanced by the needle pointer, which records the direct readings of the temperature.

**The Wanner Pyrometer.**—This instrument serves for measuring temperatures from 900 degrees Cent. and upwards. This is on similar lines to the Fery, as the temperature of a glowing body is ascertained by the intensity of light it emits. This is according to Wien's or Planck's law.

It is owing to the great increase in the intensity of light that even slight changes in temperature—tenths of a degree—give rise to perceptible and measurable differences of radiation, so that a change in the intensity of light is a delicate test respecting change in temperature.

Taking  $I$  to be the observed intensity of the rays,  $T$  the absolute temperature,  $\lambda$  the wave length of the part of the spectrum used,  $c_1$  and  $c_2$  two constants,  $e$  the basis of the nat. logarithms, the following is the equation which connects the value:—

$$I = \frac{c_1}{\lambda^5} \cdot e^{-\frac{c_2}{\lambda T}} \text{ (Wien's formula),}$$

with an essential limitation to be referred to later on. As there is no measure by means of which intensities of light can be determined so as to be scientifically correct, one can but compare the intensity of one source of light with that of another.

Assuming  $I_n$  to be this comparison measure, and  $T_n$  the corresponding absolute temperature, it follows, of course:

$$I_n = \frac{c_1}{\lambda^5} e^{-\frac{c_2}{\lambda T_n}} \text{ or}$$

$$\frac{I}{I_n} = e^{-\frac{c_2}{\lambda} \left( \frac{1}{T} - \frac{1}{T_n} \right)}$$

If one knows in this equation the value of  $I_n$  and  $T_n$ , viz., the standard measures, and, moreover, that of  $I$  and  $c_n$ , there is left only  $T$  as an unknown quantity which can be ascertained by calculation.

The aforesaid limitation is as follows:—In point of fact, the above-mentioned law applies only to so-called absolutely dark bodies. By making a small aperture into the wall, the radiation is not affected to a measurable degree; it remains, therefore, absolutely dark.

The light observed enters the apparatus through a lens, passes through lenses and a right angle prism, there is a spectrum from which, by means of a screen, light of a certain wave length is cut off, and the intensity of the light is measured by polarisation.

The part of the apparatus facing the radiation to be measured is fitted with a small 6-volt electric lamp, the light likewise passes through the apparatus and is measured in the same way of a standard with regard to the intensity of the light measured. Looking through the apparatus, one sees a circular field of view divided into two semicircles, one of which is illuminated by the small electric lamp, the other is red by the light emitted by the substance to be examined. By adjusting a movable eyepiece in which there is a Nicol prism, the two semicircles of the field in view can be adjusted to equal intensity. By the aid of the graduated circle one reads off the rotation, and by referring to a table one ascertains the temperature, which is calculated on the basis of the aforesaid law.

There are one or two very good automatic  $\text{CO}_2$  recorders on the market, and amongst the best known are (1) The "Sarco," made by Sanders, Kelders, & Co. Ltd., Fern Hill Street, London, E.C.; (2) The Simmance & Abney's Draught Recorder, by Messrs Alex. Wright & Co., Westminster. Either apparatus automatically records continuously on a chart, which is changed every twenty-four hours, the percentage of  $\text{CO}_2$  in either the boiler flues or waste flues. Both of these instruments are most suitable for continuous  $\text{CO}_2$  records in the manufacture of water gas, where such instruments are continually in use they are of benefit, and help the efficient working of the plant. The former apparatus is described on pp. 63-66, while the latter instrument is illustrated on p. 67, and referred to on p. 68.

Fig. 22 shows the general view of the "Sarco" recorder.

Fig. 23 shows the same instrument, but a section elevation view.



A ½-in. pipe, which taps the side flue or last combustion chamber of the boiler or furnace, is connected to the instrument at 3 (Fig. 25), and in order that the gas samples may

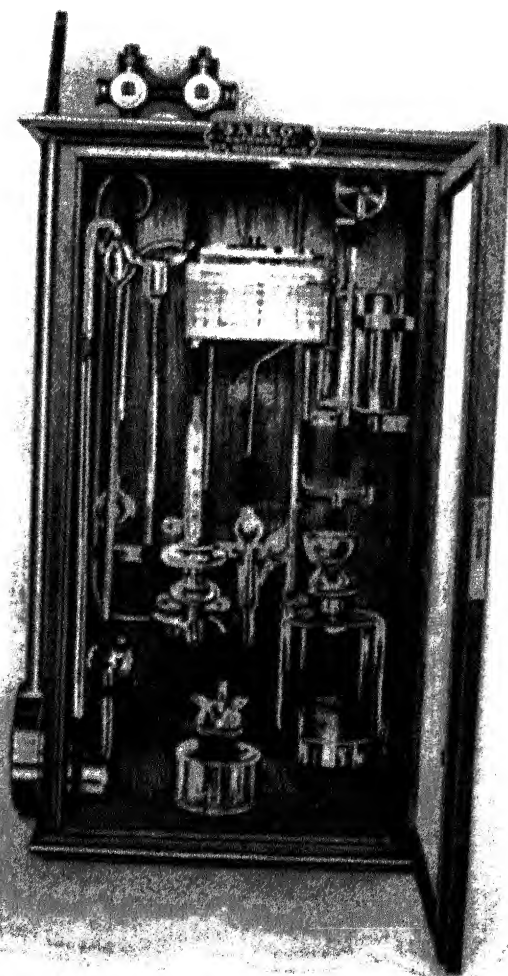
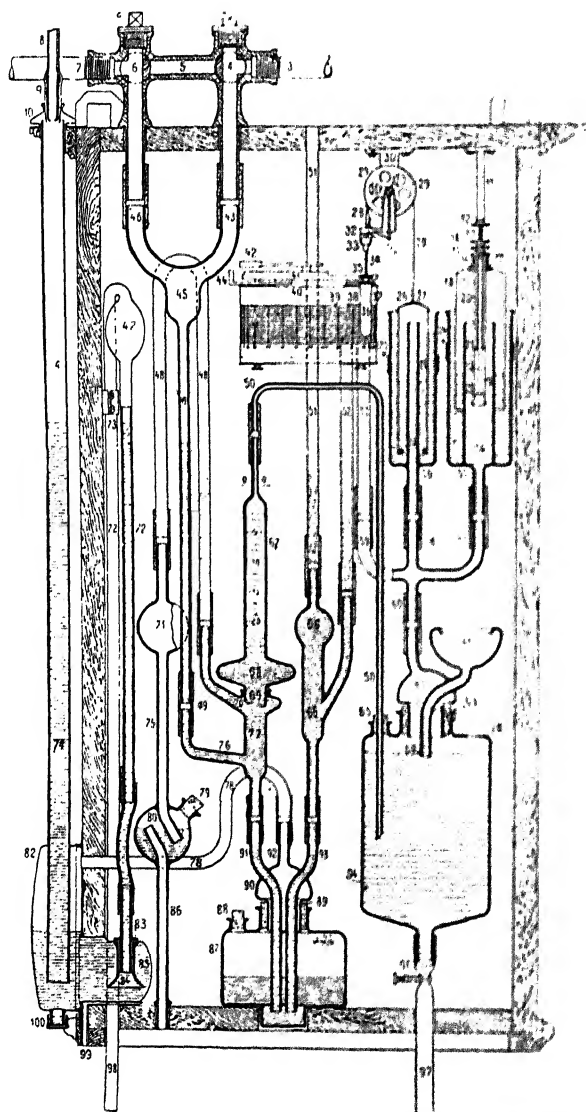


FIG. 25.—THE SARCO AUTOMATIC CO<sub>2</sub> RECORDER—GENERAL VIEW.

be secured rapidly and continuously the circuit is completed by another pipe of the same diameter. This is

FIG. 23.—SECTIONAL VIEW OF THE SARCO AUTOMATIC CO<sub>2</sub> RECORDER.

connected at 7, and carried to the base of the chimney, or, where this is impracticable, to a convenient point in the main flue, well beyond the boiler damper. Thus a continuous and rapid passage of the gas is secured, which, in average cases, renders it possible to read on the chart the effect of an alteration in the firing within two minutes of its occurrence.

The instrument is so sensitive that the slightest change is clearly depicted, and its operations are so rapid that as many as thirty-five to forty separate analyses can be recorded per hour.

The power required to procure and deal with the gas samples is derived from a fine stream of water at a head of about 2 feet. Any ordinary clean water may be used, only 2-5 gallons are required per hour (according to the speed at which the machine is operated), and the water may be used again after passing through the recorder.

It enters the instrument at 8 through the small glass injector 9. Of the latter several are provided, having apertures of various sizes, and by their use the speed of the machine may be adjusted at will. The water now flows through 74 into the power vessel 82; here it compresses the air above the water level, and this pressure is transmitted to vessel 87 through tube 78. The pressure thus brought to bear on the surface of the liquid (two parts of water to one of glycerine) with which 87 is filled to mark 95, sends the liquid upwards through tubes 91 and 93. Thence it passes up into vessels 77, 66, 67, and 68, and into tubes 51, 52, 48, and 49. Here it rises until it reaches the zero mark, which will be found on the narrow neck of vessel 67.

At the moment it reaches this mark, the power water, which, simultaneously with rising in vessel 74, has also travelled upwards in syphon 72, will have reached the top of this syphon, which then commences to flow.

Through this syphon 72 a much larger quantity of water is disposed of than flows in through injector 9, so

that the power vessels 74 and 82 are rapidly emptied again.

The moment the pressure on vessel 87 is released, the liquids return from their respective tubes into this vessel.

Presuming tube 49 to be in connection with a supply of flue gas, a sample of this is drawn in from the continuous stream which passes through 43, 45, and 46, as the liquid recedes in 49, by the vacuum which is created by the falling of the liquid.

As soon as the liquid has dropped below point 76, which is the inlet of the flue gas into vessel 68, the gas rushes up into this vessel, and a portion out into the atmosphere through outlet 70, tube 48, and seal 80.

As soon as the flow in the syphon is interrupted, vessel 82 begins to fill again, and the liquids in tubes 91 and 93 rise afresh. The gas in 77 and 68 is now forced up into tube 50, and caused to bubble right through a solution of caustic potash (sp. gr. 1.27) with which vessel 94 is filled to mark 64.

In this process any carbon dioxide ( $\text{CO}_2$ ) that may be contained in the gas is eagerly absorbed by the potash. As the gas has to pass through the potash, the absorption is rapid and complete.

The remaining portion of the sample collects in 62 and passes up through 60 into tubes 57 and 58. (It cannot pass out at 59 as this outlet is sealed by the liquid in 52.)

The gas now passes under the two floats 18 and 26, whereof the former is constructed larger and lighter, and will therefore be raised first.

By an adjustment of the thumbscrews 14 and 15 the stroke of this float is adjusted until just 20 per cent. of the whole of the sample (100 c.cm.) remains to raise float 26, when nothing is absorbed by 94. This float has attached to it pen 36, which is caused to travel downwards on the art, when 26 rises. If no  $\text{CO}_2$  was contained in the gas no gas would be absorbed by the potash in 94, and the whole of the 20 per cent. would reach float 26. Thus

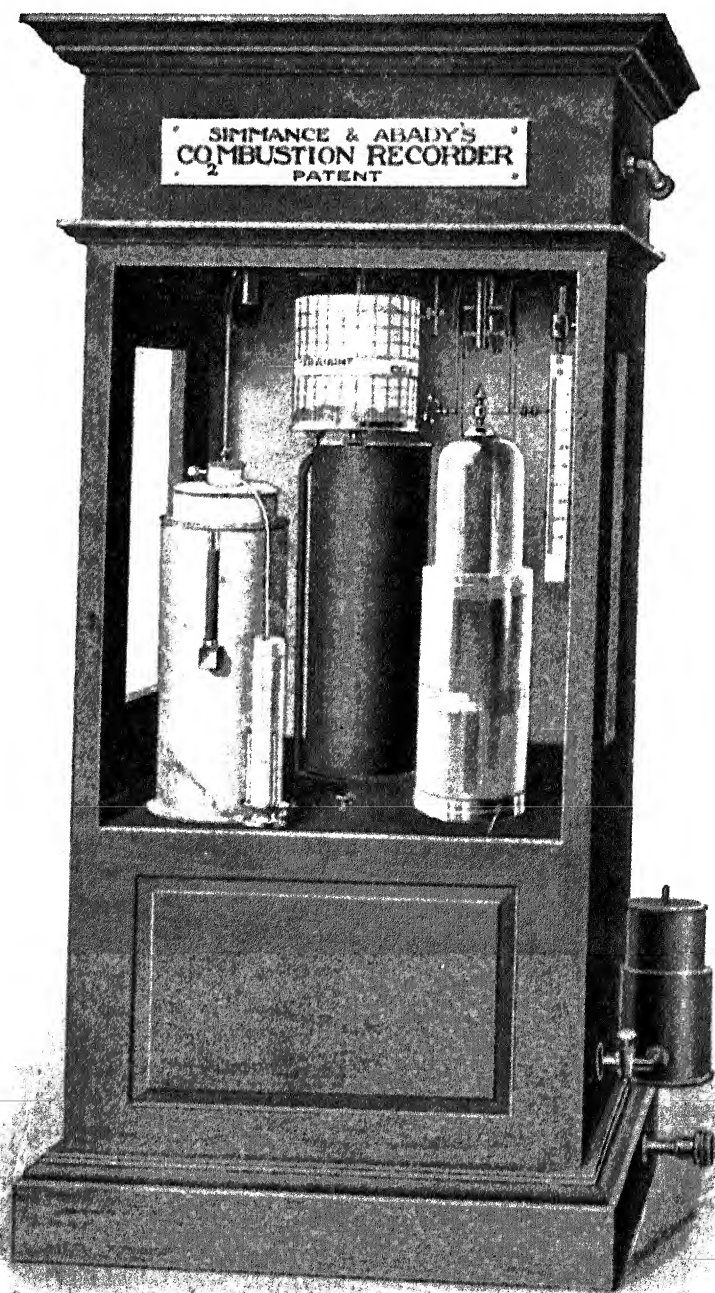


FIG. 24.—THE SIMMANC & ABADY AUTOMATIC  $\text{CO}_2$  AND DRAUGHT RECORDER.

the pen would be caused to travel the whole length of the chart from the 20 per cent. line at the top to the zero line at the bottom. Any  $\text{CO}_2$  gas contained in the sample would be absorbed by the potash, a correspondingly less quantity would reach float 26, and pen 36 would not travel right down to the bottom of the chart, *i.e.*, the zero line. Thus any  $\text{CO}_2$  absorbed will be indicated by the tops of the lines on the chart.

On the return stroke of the liquid the gas is drawn out from under the floats 18 and 26 through 57 and 58, and into tubes 59 and 52. From here it passes out into the atmosphere at 66, and through tube 51, as soon as the liquid has fallen below the outlet of tube 52.

It will be seen that the gas, when analysed, leaves the recorder by a set of tubes entirely separate from those through which the samples are obtained, so that there is no possibility of mixing the old with the new.

The Simmance-Abady  $\text{CO}_2$  Combustion Recorder, in addition to performing the continuous  $\text{CO}_2$  record, also marks continuously on the chart either the amount of draught in the boiler or main flue, or the difference between the draught below the fire and in the boiler flue, and therefore furnishes the fireman with an additional guide as to the thickness or condition of the fuel bed. It is different in construction to the "Sarco" apparatus, being entirely without glass parts or rubber tubes, while the motive power is a slight dribble of water. The whole apparatus is contained in a small iron case. It is an English-made apparatus, and is very simple in its construction, while an important point in its favour is that it is unaffected by changes in temperature. It draws the gases whether they are under forced, induced, or natural draught, and its record of  $\text{CO}_2$  is a continuous line.

## CHAPTER IV.

### *PRODUCTS OF CARBONISATION.*

OF the products of carbonisation or "destructive distillation" the chief one from a gas engineer's point of view is, of course, gas.

The yield of gas will greatly vary according to the temperature of the retort, the class of coal, the quantity carbonised, the period of distillation, and numerous other factors, known and unknown. The yield of gas per ton of coal carbonised, if the temperature is comparatively low, would be about 9,500 cub. ft., 11 gallons of tar, a low yield of ammonia as well as other bye-products, as cyanogen, naphthalene, &c. The average analysis of a gas of this kind which had been purified for the elimination of  $\text{NH}_3$ ,  $\text{CO}_2$ ,  $\text{SH}_2$ , &c., would be :—

$\text{CO}_2$	-	-	-	-	0.20 to	0.5	per cent.
CO	-	-	-	-	5.00	"	7.00 "
Methane	-	-	-	-	37.00	"	39.00 "
Hydrogen	-	-	-	-	51.00	"	53.00 "
Unsat. Hydrocarbons	-	-	-	-	7.0	"	9.0 "

The tar would be richer in its various bye-products, such as naphthalene, light oils, phenols, &c. The illuminating power would be higher, also the specific gravity and the calorific value.

As the temperature of carbonisation increases, the yield of gas increases, the make of tar both in quantity and quality drops off, and the illuminating power drops, but more ammonia is formed.

The hydrogen increases, likewise the marsh gas or methane, but the olefine or unsaturated hydrocarbons will be found to have decreased, and the naphthalene will be found to have increased.

When we get the higher heats of the up-to-date and present day carbonising, with a yield of gas of over 11,000 cub. ft. per ton, the gas will necessarily be of a much poorer quality, the illuminating power will have decidedly decreased to the extent of a candle or two. The make of tar will show a corresponding decrease, and that troublesome product naphthalene will cause more trouble than ever. It is generally known that although the specific gravity of the tar increases, the higher the make, the quantity of tar is less. The cause of this increase in gravity is due solely to the increase in pitch.

The ammonia, naphtha, and light oils decrease in quantity; the creosote, anthracene, &c., in the same direction, although in a less marked degree.

Mr Lewis T. Wright, F.C.S., has carried out some valuable experiments in this direction, and he found the effect of heat on the yield of quantity of tar is affected to a much greater extent than the gas by high heats. He found that not only was the amount of gas given off greatly augmented, but it lowered the weight of tar, though it doubled the amount of free carbon in it by specific gravity being increased.

Experimenting with a caking coal, Mr Wright found that as the make of gas increased so correspondingly did the specific gravity of tar, although the quantity showed a decrease.

The following Table shows the increases :—

Yield of Gas per ton.			Specific Gravity of Tar.
6,600 cub. ft.	-	-	1,086 degrees.
7,200 "	-	-	1,120 "
8,900 "	-	-	1,140 "
10,162 "	-	-	1,154 "
11,700 "	-	-	1,206 "



He also showed that although the percentage of free carbon is augmented, the lighter constituents in the tar are decomposed into gas.

One of the most interesting points which Mr Wright noted was the very large increase in the yield of cyanogen. At low temperature this product is yielded in very small quantities, and at a higher temperature it is found to increase by about ten times its quantity.

At this temperature the ammonia is found to have decreased, and no doubt at higher temperatures the ammonia is decomposed, and the nitrogen set at liberty combines with the free carbon forming cyanogen, whilst the excess hydrogen remains in the gas as usual.

The accompanying Table shows the decrease and increase in some of the various products on carbonising coal at various heats:—

TABLE SHOWING EFFECT OF HEAT ON GAS AND BYE-PRODUCTS.

Products.	Low Heats, 1,600° to 1,800° Fahr.	Medium Heats, 1,800° to 2,200° Fahr.	High Heats, 2,200° to 2,800° Fahr.
Gas quantity - - -	Small -	Medium -	Large.
Illuminating power -	High -	Lower -	Lowest.
Tar			
Pitch - - - -	Low -	Higher -	Highest.
Ammonia - - -	High -	Medium -	Lower.
Phenols - - -	High -	Medium -	Lower.
Ammonia - - -	Small -	Maximum	Lower.
Carbon bisulphide -	Small -	Medium -	Maximum.
Sulphuretted hydrogen -	Small -	Medium -	Maximum.
Carbon dioxide - -	Small -	Medium -	Maximum.
Cyanogen - - -	Small -	Medium -	Maximum.
Coke - - - -	Soft -	Harder -	Hardest.

Mr Lewis T. Wright gives the following interesting Table; he examined the composition of the tar, yielded by the same kind of coal, at different carbonising temperatures, between 600 and 800 degrees Cent.:—

	I.	II.	III.	IV.	V.
Cubic feet of gas- yield } per ton of coal - - }	6,600	7,200	8,000	10,162	11,700
Specific gravity of tar -	1.086	1.102	1.140	1.154	1.206
Composition of tar per cent., by weight--					
Ammoniacal liquor	1.20	1.03	1.04	1.05	0.383
Crude naphtha - -	9.17	9.65	3.73	3.45	0.095
Light oil - - -	10.50	7.46	4.47	2.59	0.567
Creosote oil - -	26.45	25.83	27.29	27.33	19.440
Anthracene oil - -	20.32	15.57	18.13	13.77	12.280
Pitch - - - -	28.89	36.80	41.80	47.07	64.080

This Table shows how the temperature of carbonisation affects not only the gas, but the composition of the bye-products are affected to an enormous extent. These results were undoubtedly got from a good coal, and would be still more marked in a poorer class of coal where the gas engineer's aim and object was to keep up his make per ton of coal. The bye-products would suffer, and undoubtedly it is possible to have the heats sufficiently high to have a tar which would be useless for working up the bye-products, as it would practically consist of pitch and ammoniacal liquor.

Lunge says on this interesting subject: Most gas engineers try above everything to get as much gas as possible out of the coal, and therefore distil at the highest possible temperature. Up to a certain point this is quite rational, and is even unavoidable from the nature of the material now universally employed for gas retorts, viz., fire-clay. This point seems to be reached when the fatty compounds are split up as far as possible before any considerable separation of free carbon has taken place.

Beyond this point more gas will be got, but its illuminating power will be less; the tar will at first contain a little more of the valuable anthracene, but at the same time even more of naphthalene, which has much less value, and of

phenanthrene, pyrene, chrysene, diphenyl, &c., which are quite valueless, so that its value will on the whole be less.

There is also a reduced quantity of tar, if the temperature in the retorts is raised higher. The separation of free carbon in the retorts and the tar is also largely increased. In England the usual temperature of working seems to be about 1,100 degrees Cent. (= 2,000 degrees Fahr.).

But, properly speaking, it should be experimentally ascertained (and that for every class of gas-coal specially) at what temperature the maximum of lighting power is obtained, even if concentrated in a smaller volume of gas, and also at what temperature we can get a maximum yield of benzene, toluene, phenols, and anthracene in the tar. Probably the two maxima will not coincide, and it will then be a matter of business calculation whether the one or the other is to be worked for. It is evident that the market prices of the bye-product must influence this consideration.

Certain statements have been made as to the shape of the retorts affecting the tar; these have evidently been wrongly interpreted, the difference in the tar being undoubtedly due to the length of time the gas hangs about in the retort, which causes decomposition of the tar and gas.

The quality of the tar now made from various kinds of cannel coal is very different from that obtained formerly. Twenty years ago, when low heats were used at the gas-works, as much as 8 per cent. of naphtha (*i.e.*, benzene and its homologues) was obtained by distillation with steam. This diminished slowly as the heat employed at the gas-works increased, until it had fallen a few years ago to about 3 per cent.

The naphtha from Scotch tar was always rich in toluene, and contained less benzene than from ordinary bituminous coal. It contains little naphthalene, and very little anthracene, so little that its extraction is not worth while. It also contains considerable quantities of paraffin, but mostly of a low melting point. Naphthalene and paraffin seem to go

together; wherever there is much of the one, the other is always present too, with few exceptions. Coal-tar is a black, more or less viscid fluid of peculiar smell, generally of phenols, sp. gr. from 1.1 to 1.2, according to the method of manufacture.

Coal-tar is an extremely complex mixture of chemical compounds, some of which have not yet been completely isolated. The tar contains nitrogenous compounds chiefly of a basic nature, owing to the nitrogen which originally exists in the coal and sulphur compounds derived from pyrites, &c., which are always found in the coal.

James M'Leod, in the *Journal Soc. Chem. Ind.*, vol. xxvi., p. 137, says that on submitting coal to destructive distillation, the nitrogen present in it is partly retained by the coke, and partly eliminated as free nitrogen, which at the moment of liberation probably combines with hydrogen and with carbon to form ammonia and cyanogen respectively, and partly remains as free nitrogen. It also remains combined with carbon and nitrogen to form bases, such as pyridine.

The summary of his results is:—

	Per Cent. of Total Nitrogen.
Nitrogen in the coke	58.30
" " tar	3.90
" " ammoniacal liquor	17.10
" " cyanogen	1.20
" " in gas (by difference)	19.50
	<hr/> 100.00 <hr/>

He also gives the average nitrogen in coal, 1.434 per cent., in coke from coal, 1.374 per cent., which gives the total nitrogen in the coke 58.3 per cent.

Lunge gives the following list of compounds hitherto found in coal-tar, or reasonably presumed to exist in it:—

## A. HYDROCARBON.

I. *Methane Series*,  $C_nH_{2n+2}$ .

Formula	Melting Point	Boiling Point
Deg. Cent.	Deg. Cent.	
Methane . . . . .	$CH_4$	
Ethane . . . . .	$C_2H_6$	
Propane . . . . .	$C_3H_8$	-29
Butane (normal) . . . . .	$C_4H_{10}$	-1
Pentane (normal) . . . . .	$C_5H_{12}$	liquid 37-39
Isopentane . . . . .	$C_5H_{12}$	-49
Hexane (normal) . . . . .	$C_6H_{14}$	60-77
Heptane (normal) . . . . .	$C_7H_{16}$	98
Ethylmethyl . . . . .	$C_7H_{16}$	90-4
Octane I. . . . .	$C_8H_{18}$	110-120
" II. . . . .	$C_8H_{18}$	124
Nonane I. . . . .	$C_9H_{20}$	110
" II. . . . .	$C_9H_{20}$	130-8
Decane I. . . . .	$C_{10}H_{22}$	158-161
" II. . . . .	$C_{10}H_{22}$	170-171
Undecane . . . . .	$C_{11}H_{24}$	180-182
Dodecane . . . . .	$C_{12}H_{26}$	200-202
Tridecane . . . . .	$C_{13}H_{28}$	218-220
Quatordecane . . . . .	$C_{14}H_{30}$	236-240
Quinderane . . . . .	$C_{15}H_{32}$	256-262
Sesterane . . . . .	$C_{16}H_{34}$	280
Solid paraffins . . . . .	$\left. \begin{array}{l} C_{17}H_{36} \\ \text{to} \\ C_{27}H_{56} \end{array} \right\}$	40-60

II. *Ethylene Series*,  $C_nH_{2n}$ .

Ethylene . . . . .	$C_2H_4$	110
Propylene . . . . .	$C_3H_6$	"
Butylene (normal) . . . . .	$C_4H_8$	6
Pseudobutylene . . . . .	$C_4H_8$	1
Isobutylene . . . . .	$C_4H_8$	8
Amylene . . . . .	$C_5H_{10}$	liquid 39
Hexylene . . . . .	$C_6H_{12}$	68-70
Heptylene . . . . .	$C_7H_{14}$	96-99

III. *Hexahydro-addition-products of the Benzene Series*,  $C_{10}H_{16}$  (Naphthenes).

Hexahydrobenzene . . . . .	$C_{10}H_{16}$	liquid 60
Hexahydrotoluene . . . . .	$C_{11}H_{18}$	97
Hexahydroisoxylene . . . . .	$C_{11}H_{18}$	118

	Formula.	Melting Point.	Boiling Point.
		Deg. Cent.	Deg. Cent.
A. HYDROCARBONS-- <i>contd.</i>			
IV. <i>Acetylene Series</i> , $C_nH_{2N-2}$ .			
Acetylene . . . . .	$C_2H_2$	...	...
Allylene . . . . .	$C_3H_4$	...	...
Crotonylene . . . . .	$C_4H_6$	liquid	18
Valylene (?) . . . . .	$C_5H_8$	...	...
Hexoylene . . . . .	$C_6H_{10}$	liquid	80
Higher members . . . . .	$C_{12}H_{22}$	"	210
" " . . . . .	$C_{14}H_{24}$	"	240
" " . . . . .	$C_{16}H_{26}$	"	280
V. <i>Tetrahydro - addition - products of the Benzene Series</i> , $C_nH_{2N-2}$ (Naphthylenes).			
Tetrahydrobenzene . . . . .	$C_6H_{10}$	liquid	82
Tetrahydrotoluene . . . . .	$C_7H_{12}$	"	103-105
Tetrahydroxylene . . . . .	$C_8H_{14}$	"	120-132
VI. <i>Series</i> $C_nH_{2N-4}$ .			
Cyclopentadiene . . . . .	$C_5H_6$	liquid	42-5
Nonone . . . . .	$C_9H_{14}$	"	174
VII. <i>Aromatic Dihydro - addition - products</i> , $C_nH_{2N-4}$ (Terpenes).			
Dihydrobenzene . . . . .	$C_6H_8$	liquid	81.5
Dihydrotoluene . . . . .	$C_7H_{10}$	"	105-108
Dihydroxylene . . . . .	$C_8H_{12}$	"	132-134
Dihydrocymene . . . . .	$C_{10}H_{16}$	"	174
VIII. <i>Benzene Series</i> , $C_nH_{2N-6}$ .			
Benzene . . . . .	$C_6H_6$	4-5-7	80.4
Toluene . . . . .	$C_7H_8$	liquid	111
Xylene . . . . .	$C_8H_{10}$	"	...
Orthoxylene . . . . .	...	"	141-142
Metaxylene . . . . .	...	"	139
Paraxylene . . . . .	...	15	137.5-138
Ethylbenzene . . . . .	$C_8H_{10}$	liquid	137
Pseudocumene . . . . .	$C_9H_{12}$	"	160.5
Mesitylene . . . . .	$C_9H_{12}$	"	161
Hemellithol . . . . .	$C_9H_{12}$	"	175
Durene . . . . .	$C_{10}H_{14}$	80-81	196
Other tetramethyl benzenes . . . . .	...	...	...
IX. <i>Styrolene</i> (?), $C_nH_{2N-8}$ .			
Hydride of styrolene (?) . . . . .	$C_8H_8$	liquid	145
	$C_8H_{10}$	"	...

	Formula.	Melting Point. Deg. Cent.	Boiling Point. Deg. Cent.
A. HYDROCARBONS— <i>contd.</i>			
X. <i>Indene</i> , $C_{10}H_{10}$ . . .	$C_9H_8$	liquid	177-178
Hydrindene . . .	$C_9H_{10}$	"	...
XI. <i>Naphthalene</i> , $C_{10}H_{10}$ . . .	$C_{10}H_8$	79	218
Naphthalene dihydride . . .	$C_{10}H_{10}$	liquid	200-210
" tetrahydride . . .	$C_{10}H_{12}$	"	190
$\alpha$ -Methylnaphthalene . . .	$C_{11}H_{10}$	"	240-243
$\beta$ -Methylnaphthalene . . .	$C_{11}H_{10}$	32.5	241-242
Dimethylnaphthalene . . .	$C_{12}H_{12}$	liquid	262-264
XII. <i>Acenaphthene</i> , $C_{12}H_{10}$ . . .	$C_{12}H_{10}$	95	277.5
Acenaphthene hydride . . .	$C_{12}H_{12}$	...	260
Diphenyl . . .	$C_{12}H_{10}$	70.5	254
XIII. <i>Fluorene</i> , $C_{13}H_{10}$ . . .	$C_{13}H_{10}$	113	295
XIV. <i>Anthracene</i> , $C_{14}H_{10}$ . . .	$C_{14}H_{10}$	213	360
Anthracene dihydride . . .	$C_{14}H_{12}$	106	305
" hexahydride . . .	$C_{14}H_{16}$	63	290
" perhydride . . .	$C_{14}H_{24}$	88	250
Methylanthracene (?) . . .	$C_{15}H_{12}$	208-210	above 260
Dimethylanthracene (?) . . .	$C_{16}H_{14}$	224-225	...
Phenanthrene . . .	$C_{14}H_{10}$	99-100	340
Phenanthrene tetrahydride . . .	$C_{14}H_{14}$	liquid	300-304
" octohydride . . .	$C_{14}H_{18}$	"	...
" perhydride . . .	$C_{14}H_{24}$	- 3	270-275
Pseudophenanthrene (?) . . .	$C_{15}H_{12}$	115	above 360
Synanthrene (?) . . .	$C_{14}H_{10}$	189-195	...
Fluoranthrene (?) . . .	$C_{15}H_{10}$	109	above 360
Pyrene . . .	$C_{16}H_{10}$	148	above 360
Chrysene . . .	$C_{18}H_{12}$	250	436
Chrysene hydride . . .	$C_{18}H_{28}$	liquid	360
" perhydride . . .	$C_{18}H_{30}$	115	353
Chrysogene . . .	...	280-290	...
Retene . . .	$C_{18}H_{18}$	98-99	350
Retene dodecahydride . . .	$C_{18}H_{30}$	liquid	336
Succisterene (?) . . .	...	160-162	above 300
Picene . . .	$C_{20}H_{14}$	364	518-520
Picene eicosylhydride . . .	$C_{20}H_{34}$	liquid	360
" perhydride . . .	$C_{20}H_{36}$	175	360
Benzerythrene . . .	$C_{24}H_{18}$	307-308	...
Bitumene . . .	...	...	...

	Formula.	Melting Point.	Boiling Point.
		Deg. Cent.	Deg. Cent.
B. OXYGENISED COMPOUNDS.			
Water . . . . .	$H_2O$	0	100
Methylic alcohol (?) . . . . .	$CH_3O$	liquid	63
Ethylic alcohol (?) . . . . .	$C_2H_5O$	"	78.5
Acetone . . . . .	$C_3H_6O$	"	56
Ethylmethylketone (?) . . . . .	$C_4H_8O$	"	77.5-81
<i>Acids and Phenols.</i>			
Acetic acid . . . . .	$C_2H_4O_2$	16	119
Benzoic acid . . . . .	$C_7H_6O_2$	121	249
Phenol (carbolic acid) . . . . .	$C_6H_6O$	42	184
Orthocresol . . . . .	$C_7H_8O$	12	188
Paracresol . . . . .	"	36	199
Metacresol . . . . .	"	34	201
Xylenols : ortho 1, 2, 4 . . . . .	$C_8H_{10}O$	62	225
meta 1, 2, 3 . . . . .	"	73	216
meta 1, 3, 4 . . . . .	"	26	211.5
para 1, 3, 4 . . . . .	"	74.5	211-213
$\alpha$ -Naphthol . . . . .	$C_{10}H_8O$	94-96	278-280
$\beta$ -Naphthol . . . . .	"	122	294
<i>Phenols of the Anthracene Series (?).</i>			
$\alpha$ -Pyrocresol . . . . .	$C_{15}H_{14}O$	106	350
$\beta$ -Pyrocresol . . . . .	"	124	"
$\gamma$ -Pyrocresol . . . . .	"	105	"
Rosolic acid (?) . . . . .	$C_{19}H_{14}O_3$	"	"
Brunolic acid (?) . . . . .	"	"	"
Cumarone . . . . .	$C_9H_8O$	liquid	108.5-109.5
<i>p</i> -Methyl-cumarone . . . . .	$C_{10}H_8O$	"	197-199
<i>m</i> -Methyl-cumarone . . . . .	"	"	195-196
<i>o</i> -Methyl-cumarone . . . . .	"	"	190-191
<i>o.p.</i> -Dimethyl-cumarone . . . . .	$C_{10}H_{10}$	"	221-222
<i>m.p.</i> -Dimethyl-cumarone . . . . .	"	"	221
<i>o.m.</i> -Dimethyl-cumarone . . . . .	"	"	216
C. SULPHURETTED COMPOUNDS.			
Hydrogen sulphide . . . . .	$H_2S$	...	...
Ammonium sulphide . . . . .	$(NH_4)_2S$	...	...
" sulphocyanide . . . . .	$(NH_4)CNS$	...	...
Sulphur dioxide . . . . .	$SO_2$	...	...
Carbon bisulphide . . . . .	$CS_2$	liquid	47
" oxysulphide . . . . .	$COS$	...	...
Mercaptanes . . . . .	...	...	...
liol (?) . . . . .	...	...	...
phene . . . . .	$C_4H_4S$	liquid	48



	Formula.	Melting Point.	Boiling Point.
		Deg. Cent.	Deg. Cent.
C. SULPHURETTED COMPOUNDS —continued.			
$\alpha$ -Thiotolene . . . .	$C_5H_6S$	liquid	113
$\beta$ -Thiotolene . . . .	"	"	113
$\alpha$ - $\alpha$ -Thioxene . . . .	$C_6H_6S$	"	135-136
$\alpha$ - $\beta$ -Thioxene . . . .	"	"	136-137
$\alpha$ - $\beta^1$ -Thioxene . . . .	"	"	137-138
$\beta$ - $\beta$ -Thioxene . . . .	"	"	136-137
$\alpha$ - $\beta$ - $\beta^1$ -Trimethylthiophene . . . .	$C_7H_{10}S$	"	163
Tetramethylthiophene . . . .	$C_8H_{12}S$	"	182-184
Biophen . . . .	$C_4H_4S_2$	"	165-170
$\alpha^1$ - $\alpha$ -Dithienyl . . . .	$C_8H_6S_2$	33	...
$\beta^1$ - $\beta$ -Dithienyl . . . .	"	132.4	...
Trithienyl . . . .	$C_{12}H_8S_3$	147	357
Thionaphthene . . . .	$C_8H_6S$	30-31	220-221
Thiophthene . . . .	$C_6H_4S_2$	liquid	224-226
D. CHLORINATED COMPOUNDS.			
Ammonium chloride . . . .	$NH_4Cl$	...	...
E. NITROGENISED COMPOUNDS.			
I. Basic.			
Ammonia . . . .	$NH_3$	...	...
(Ammonium compounds mentioned under C., D., and E., II.)	...	...	...
Methylamine, ethylamine, &c. . . .	...	liquid	...
Cespitine (?) . . . .	$C_5H_{13}N$	"	95
Aniline . . . .	$C_6H_7N$	-8	182
Homologues of aniline (?) . . . .	...	...	...
Pyridine . . . .	$C_5H_5N$	liquid	116.7
$\alpha$ -Picoline . . . .	$C_6H_7N$	"	135
$\gamma$ -Picoline . . . .	"	"	(?)
$\alpha$ - $\alpha$ -Lutidine . . . .	$C_7H_9N$	"	142
$\alpha$ - $\gamma$ -Lutidine . . . .	"	"	157
$\alpha$ - $\beta_1$ -Lutidine . . . .	"	"	(?)
$\beta$ - $\gamma$ -Lutidine . . . .	"	"	163.5-164.5
$\beta$ - $\beta_1$ -Lutidine . . . .	"	"	169-170
$\gamma$ -Ethylpyridine . . . .	"	"	164-166
$\alpha$ - $\gamma$ - $\alpha^1$ -Collidine . . . .	$C_8H_{11}N$	"	171-172
$\alpha^1$ - $\beta$ - $\gamma$ -Collidine . . . .	"	"	165-168
Parvoline (?) . . . .	$C_9H_{13}N$	"	188
$\alpha$ - $\beta$ - $\gamma$ - $\beta^1$ -Tetramethylpyridine . . . .	"	"	232-234
Coridine (?) . . . .	$C_{10}H_{15}N$	"	211
Rubidine (?) . . . .	$C_{11}H_{17}N$	"	230

	Formula.	Melting Point.	Boiling Point.
		Deg. Cent.	Deg. Cent.
E. NITROGENISED COMPOUNDS —continued.			
I. Basic—continued.			
Viridine (?) . . . . .	$C_{12}H_{19}N$	liquid	251
Leucoline (Chinoline) . . . . .	$C_9H_7N$	„	239-240
Isoquinoline . . . . .	$C_9H_7N$	18-23	236-237
Chinaldin ( $\alpha$ -Methyl-quinoline) . . . . .	$C_{10}H_9N$	liquid	243
Iridoline ( $\gamma$ -Methyl-lepidine) . . . . .	$C_{10}H_9N$	„	252-257
Cryplidine (Dimethyl-quinoline) . . . . .	$C_{11}H_{11}N$	„	274
Tetracoline-octacoline (?) . . . . .	„	„	„
Acridine . . . . .	$C_{13}H_9N$	111	above 360
2-Methylacridine . . . . .	$C_{14}H_{11}N$	134	„
4-Methylacridine . . . . .	„	88	„
2-4-Dimethylacridine . . . . .	$C_{15}H_{13}N$	71	„
II. Not Basic.			
Pyrrol . . . . .	$C_4H_5N$	liquid	133
Ammonium cyanide . . . . .	$CN.NH_4$	„	„
Methylic cyanide (acetonitrile) . . . . .	$CH_3-CN$	liquid	77
Benzonitrile . . . . .	$C_6H_5CN$	„	191
Methylic isocyanide . . . . .	$C_2H_3N$	„	59.6
Carbazol . . . . .	$C_{12}H_9N$	238	355
Phenyl- $\alpha$ -naphthyl-carbazol . . . . .	$C_{16}H_{11}N$	225	above 440
Phenyl- $\beta$ - „ „ . . . . .	„	230	„
F. FREE CARBON.	Cx	„	„

It is sometimes necessary to test a sample of tar in the laboratory to ascertain its market value. The following is the usual method, devised by Lunge, and given in his "Coal-Tar and Ammonia" :—

In a scientific laboratory it is difficult to employ more than a kilogram or two for each distillation. The results thus obtained will never exactly coincide with those obtained in manufacturing practice, but experience proves that they give a very good idea of the general quality of the tar. It would be most convenient to employ for such quantities distilling vessels made of metal, and I would indeed strongly recommend this for factory laboratories; all the more as it is

thus easy to work upon very much larger quantities. But in scientific laboratories where, for more reasons than one, it is out of the question to distil a hundredweight of tar, or some such quantity, it will be always preferred to accurately observe the progress of the operation, and this it is only possible to do in glass retorts.

My retorts were tubulated, holding about 5 litres, and were heated in a kind of sand air bath—that is, in a suitably-shaped thin wrought-iron dish, the bottom of which was covered by a layer of sand 1 cm. thick. About half

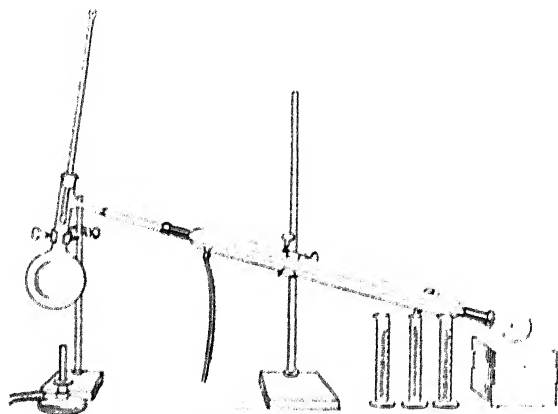


FIG. 20.—APPARATUS FOR DISTILLING A SAMPLE OF TAR.

of the retort was within the dish, and the whole of it, down to the sand, and including the upper part of the neck, was wrapped round with wire gauze. The heating was done by a Fletcher's gas stove, placed in a large flat pan partially filled with sand. Hence, in case of an accident, the tar would have first to run into the upper pan, forming the sand air bath, and anything boiling over from this would have been caught in the lower pan. The tubulure of the retort was fitted with a twice perforated cork, holding a thermometer and a tube, drawn out into a capillary at the lower end, with the object of passing a minute current of

air bubbles through the liquid, in order to prevent bumping. This precaution, first introduced by Dittmar, and also employed by Watson Smith, was found to be very useful indeed, but it seems possible that the air current might carry away a minute quantity of benzene.

The retort was, during the first part of the operation, connected with a Liebig's condenser, so long as the distillate remained entirely liquid. When it began to partly solidify—that is, between 170 and 180 degrees—the cooler was removed; and since now the last portions of water had been volatilised, and no more bumping was to be apprehended, the current of air was discontinued. The last of the water escaped between 140 and 170 degrees with explosive violence.

The distillation of  $2\frac{1}{2}$  to 3 litres of tar took about eight hours. It is decidedly advisable to carry the distillations through without any interruption, both because the heating up, after the contents of the retort have been semi-solid or solid on cooling, is always an awkward operation; and because during the cooling down and the heating up a considerable quantity of substance passes over far below their proper boiling points.

The distillates were collected in tared, narrow, graduated cylinders, and after cooling down they were measured and weighed.

The fractions were made in the way stated below. But although, as a matter of course, every precaution was taken to keep the temperature as constant as possible, still, without any recognisable reason, the thermometer showed considerable oscillations, and sometimes went down to 20 degrees without any diminution in the rate of distillation.

It cannot be said that such assays are exact analytical operations. The fractions will differ to some extent, as the distillation proceeds more or less slowly. Each time, when substances are poured from one vessel to another, small losses are unavoidable, although in the case of the higher boiling substances the vessels were rinsed with ether, which

was subsequently evaporated. In washing and drying, in the case of the first distillates also by evaporation, small losses will occur, which become all the more important when the absolute quantity of substance is only slight.

If the tar has not been previously dehydrated, the work must begin with that operation, which is of great importance. It is not feasible to go as far as 100 degrees, because then the tar would lose many valuable portions, especially as the operation takes so much time. Hence, the dehydration was performed in the retort itself, turning its neck upwards, and connecting it with a cooler, inclined downwards, in order to collect any benzene escaping along with the water. The heating was continued in this manner to 60 to 70 degrees for a full fortnight; every morning, before recommencing, the water collected in the meantime on the surface was removed by a pipette. For all that, some water remained behind, evidently in chemical combination with phenol, pyridine, &c., and this could only be removed by distillation.

The fractions were made as follows:—

1. Light oil, up to 170 degrees.
2. Middle oil, up to 230 degrees (carbolic oil).
3. Creosote oil, up to 270 degrees.
4. Anthracene oil, up to the close of the distillation, which was continued as long as anything would come over; this explains why the pitch was extraordinarily hard.

The above fractions were treated in the following manner:—The light oil was first agitated with caustic soda solution of sp. gr. 1.1, and the contraction of volume was calculated as "phenols." The oil was then washed with water, with concentrated sulphuric acid, and again with water, and the total contraction was calculated as "loss by washing." The residual oil was distilled, and the fractions coming over up to 100 and 140 degrees were separately collected. The distillate up to 140 degrees was considered as "crude aniline benzol," and its degree of purification examined by nitrification with ordinary mixture of acids.

The portion remaining behind at 140 degrees was calculated as "heavy naphtha"; it must, of course, leave a good deal of residue on rectification, and this residue will practically go to the creosote oil; but, on the other hand, some heavy naphtha will come back from the "middle oil," and on the small scale it was impossible to say how far this would compensate for the residue left on rectifying. The small quantity of liquid also made it impossible to separate the "aniline benzol" into benzene, toluene, and xylenes. From the middle oil and the creosote oil a quantity of naphthalene crystallised on cooling. This was filtered through calico, strongly pressed, and calculated as "crude naphthalene." The liquid portion of the oil (making allowance for the mechanical loss in pressing) was treated with caustic soda solution, and the contraction of volume again set down as "phenols."

The anthracene oil, after cooling, was filtered through calico, the crude anthracene was pressed cold, then spread out upon porous earthenware slabs, heated in an air bath to 30 to 40 degrees, pressed while warm, and weighed. It was now analysed by the "Höchst" test; but since in the trade anthracene is usually sold as 30 or 40 per cent., three times the weight of pure anthracene was deducted from the weight of crude anthracene oil, to get at the figure for liquid anthracene oil.

The pitch was tested for its softening point by heating a piece the size of a pea on a wire in an air bath beside a thermometer, until, by pressing with the fingers, it proved to be distinctly plastic.

The water bath did not suffice for this purpose. It was further tested for "carbon," one of the most tedious parts of the work. For this purpose it was extracted alternately with boiling benzene and carbon disulphide, but it took many days' toil. The solvents did not show any but a faint colour, and left no more residue when evaporated on a watch glass.

This operation must be carried out with the greatest

caution, since otherwise fine particles of carbon will pass through the filter; for this reason Soxhlet's extracting apparatus, otherwise so convenient, could not be employed.

The specific gravity of tar cannot be estimated by means of an ordinary specific-gravity bottle, which is too difficult to fill exactly and to clean in this case. Lunge employed a "weighing bottle" of the shape shown in Fig. 26, with a glass stopper provided with a rill *a*, 2 mm. wide. The operation is performed, as when estimating the specific gravity of solids, by filling the glass only partially with tar and then completely with water. First the glass is weighed empty (*a*), and again after being filled with water at 15 degrees Cent. (*b*). It is then dried, tar is poured in up to about two-thirds of its height, and the glass without its stopper is placed for about an hour in hot water till all air bubbles disappear. After cooling, the weight of the glass plus the tar is determined (*c*). Now water is poured in, the stopper is inserted, the water issuing from the rill is removed, the whole is allowed to stand in a vessel filled with water of known temperature, and the weight is again taken (*d*). The specific gravity (*S*) sought is:—

$$S = \frac{c - a}{b + c - (a + d)}$$

In most cases the specific gravity of tar, after dehydration, is a sufficient guide as to its quality.

According to Köhler (*Zsch. f. angew. Ch.*, 1888, p. 677), it depends mainly upon the percentage of free carbon as shown in the following Table:—

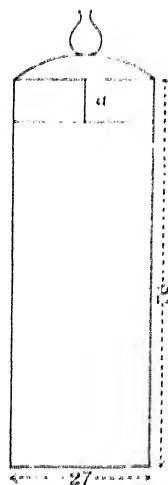


FIG. 26.  
SPECIAL SPECIFIC GRA-  
VITY BOTTLE FOR TAR.

Origin of Tar.	Specific Gravity.	Free Carbon.	Origin of Tar.	Specific Gravity.	Free Carbon.
		per cent.			per cent.
Heidelberg -	1.220	23.75	Caunstadt -	1.164	14.05
Darmstadt -	1.215	20.93	Roltweil -	1.161	14.00
Baden Baden	1.195	19.92	Karlsruhe -	1.155	13.50
Bockenheim-	1.190	18.24	Ulm -	1.150	12.44
Frankfort -	1.180	15.70	Heilbronn -	1.150	12.42
Bamberg -	1.175	15.15	Oos -	1.145	5.00
Neustadt -	1.172	15.07			

The estimation of free carbon in tar is decidedly important. Kraemer extracts the tar with forty times its weight of xylene. It is more expeditious to heat 10 grams of tar with a mixture of 25 grams glacial acetic acid, and 25 grams of toluene, pouring the liquid on to two filters of equal weight, placed one within the other, and washing with boiling benzene until this runs off colourless. After drying, the outer filter is used as tare in weighing the inner.

The more free carbon, the more viscous the tar, and the more easily will it froth during distillation. Tars containing less free carbon, that is of less specific gravity, are richer in benzene and other light hydrocarbons than those containing more free carbon. But this holds good only to a certain limit, say 15 to 17 per cent. free carbon. Above this tars of equal percentage of free carbon may furnish either more anthracene, or more benzene, &c., according to their origin; but if they contain considerably more carbon than 17 per cent., they are sure to yield less valuable products of all kinds and more pitch. If  $K$  is the percentage of free carbon in the pitch, and  $k$  the percentage of free carbon in the tar, the yield of pitch is  $\frac{100k}{K}$ . Medium hard pitch contains about 28 per cent. free C; for normal tar with 16 per cent. C we thus find:—

$$\frac{100 \times 16}{28} = 57 \text{ per cent.},$$



which is in sufficient agreement with experience. It is not proposed here to enumerate all the tests and analyses of the various compounds, &c., in tar; for further and complete information, Lunge, "Coal-Tar and Ammonia," must be consulted together with recent literature on the subject.

**Höchst Test for Anthracene.**—1 gram of anthracene cake is dissolved in 45 c.c. of glacial acetic acid in a half litre flask, to the mouth of which an inverted condenser is attached. The solution is boiled, and to this is added from a tap funnel (fitted to the top of the inverted condenser) a solution of 15 grams of chromic acid in 10 c.c. of glacial acetic acid, diluted with its own volume of water. The contents of the flask must be kept in gentle ebullition, and the chromic acid must be added drop by drop, the operation taking about two hours. The liquid in the flask must then be kept boiling for two hours longer, when the heat is removed, and the flask and contents left at rest for twelve hours. 400 c.c. of water are now added, and after leaving for three hours, the contents of the flask are filtered; the anthraquinone collected on the filter is washed in turn with cold water, boiling dilute solution of caustic potash (about 2 per cent.), and then with hot water.

The anthraquinone is transferred by washing from the filter to a porcelain dish, and then dried at 100 degrees Cent. Ten times its weight of fuming sulphuric are now added, the dish is heated on a hot-water bath for ten minutes, and then placed in a moist atmosphere for twenty-four hours; 200 c.c. of water are now added, filter, and wash mass on filter as before. The contents of the filter are transferred



FIG. 27. — HÖCHST  
TEST FOR AN-  
THRACENE.

to a dish which is dried at 100 degrees Cent. and weighed. The dish is then heated to drive off the anthraquinone, the dish is cooled, and again weighed; the difference between the two weighings gives the amount of anthraquinone obtained from 1 gram of the anthracene cake. Multiplying this by 85.58 gives the percentage of anthracene found in the 1 gram of cake; this is easily calculated into the percentage of anthracene in the tar.

**Benzene.**—The methods and procedure are too numerous to mention, but the general distillation of benzene for commercial purposes is carried out in an ordinary retort, with Liebig condenser attached. The great cause of different results is where to place the bulb of the thermometer which is generally in the liquid. G. E. Davies finds in 90 per cent. and  $\frac{50}{100}$  per cent. benzole:—

	90 per cent.	$\frac{50}{100}$ per cent.
Pure benzene	75	50
„ toluene	24	40
„ xylene	1	10

*Estimation of Sulphur in Benzole.*—A weighed quantity of the benzole is burnt on the gas referee's apparatus. A good method is: fill an ordinary spirit lamp with benzole and weigh it, place it in the trumpet tube, in the position usually occupied by the Bunsen burner, placing the usual carbonate (sesqui) of ammonia round inside the tube, and light the lamp, taking care that it is not too high and that it does not smoke. After finish of test wash down the apparatus, boil liquor with hydrochloric acid, and precipitate sulphur as  $\text{BaSO}_4$ . Weigh lamp to ascertain the amount of benzole burnt. The weight of  $\text{BaSO}_4$  multiplied by .13734 gives the amount of sulphur in quantity of benzole used.

Mr W. Irwin before the Chemical Industry in 1901 gave the above method, but he mixed a certain portion of alcohol or methylated spirit with it, first of all carrying

out a blind test to ascertain the amount of sulphur in the methylated spirit. The amount of sulphur in benzole varies from about 0.40 per cent. to 1.00 per cent. Undoubtedly in a very short time a market will be opened for a rectified benzole for the use of internal combustion motors, as the petrol used now for motor cars, &c., is not such a very great difference from ordinary benzole.

**Ammonium Sulphate.**—It is not intended to treat of the manufacture of sulphate of ammonia, but only the analysis of this material after manufacture. Sulphate of ammonia is generally tested for (1) percentage of water at 100 degrees Cent., (2) the percentage of  $\text{NH}_3$ .

The Fertilisers and Feeding Stuffs Act, 1906, Chapter 27, states: "Every person who sells for use as a fertiliser of

the soil any article which has been subjected to any artificial process in the United Kingdom, or which has been imported from abroad, shall give the purchaser an invoice stating the name of the article, and what are the respective percentages (if any) of nitrogen, soluble phosphates, insoluble phosphates, and potash contained in the

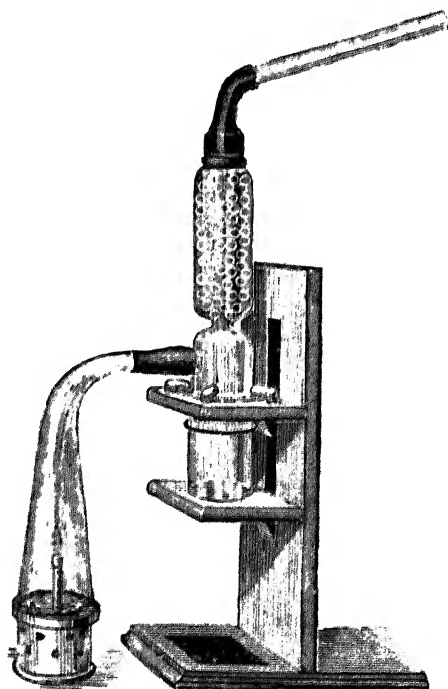


FIG. 93. APPARATUS FOR ESTIMATION OF SULPHUR IN BENZOL.

article, and the invoice shall have effect as a warranty by the seller that the actual percentages do not differ from those stated in the invoice beyond the prescribed limits of error."

The Act further gives power to appoint analyst and sampler, power to have fertiliser or feeding stuff analysed, &c. &c.

1. *Estimation of Moisture.*—The amount of free moisture is determined by drying a weighed quantity to 110 degrees Cent. until weight is constant, in a flat dish. The loss in weight represents the moisture in quantity taken.

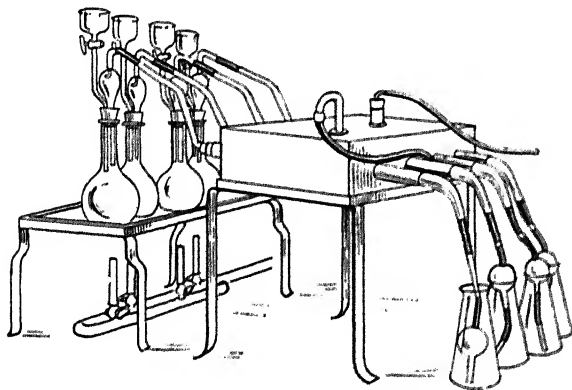


FIG. 29.—APPARATUS FOR ESTIMATION OF AMMONIA.

In sampling ammonium sulphate special care must be taken. It must be taken quickly, intimately mixed, and immediately placed in a well-stoppered bottle, so that no loss of moisture takes place.

2. *Estimation of Ammonia.*—A known weight of the salt is taken (say about 10 grams), and dissolved in 500 c.c. of distilled water; 50 c.c. of this solution are distilled with caustic potash or soda, and the vapour evolved passed into 15 c.c. of normal sulphuric acid.

Supposing that on titration 3 c.c. of acid are found to have remained unsaturated, then 12 c.c. of acid have been

neutralised. As 50 c.c. of the solution corresponds to 1 gram of original liquid (as exactly 10 grams were taken), it therefore contains  $100 \times 0.017 \times 12 = 20.4$  per cent. of ammonia. From this the nitrogen can easily be calculated.

If many of these tests are required I find that a good method to do a number at a time is to adapt Kjeldahl's apparatus for the determination of nitrogen, consisting of six flasks, copper condensers, and flasks on outlet (as per sketch). This apparatus can either be purchased with horizontal or upright condensers, and takes up very little room.

## CHAPTER V.

### *ANALYSIS OF CRUDE COAL-GAS.*

THE impurities in coal after leaving the condensers are as follows :—

Ammonia, about	-	-	300	grains per cubic foot.
Carbonic acid	-	-	1,800	" "
Sulphuretted hydrogen	-	-	1,050	" "
Carbon disulphide	-	-	70	" "
Other sulphur compounds	-	-	10	" "
Cyanogen as hydrocyanic acid	-	-	106	" "

For the efficient working of a gasworks it is necessary to know the amount of these various impurities, and often at various parts of the plant. It is not proposed to deal with any part of the plant, so only methods of analysing or testing for these impurities will be given.

Taking them in the order mentioned :—

**Ammonia.**—A normal solution of sulphuric acid is made up as described in Chapter I., and likewise a normal solution of caustic soda. These are titrated against each other. 200 c.c. of the normal sulphuric are taken and put into a couple of Woulfe bottles after adding a few drops of methyl orange, which gives a yellow colour to the liquid. The gas is now bubbled through the acid and then through a meter. It is generally advisable to have a third bottle with a little distilled water in it which has also had a few of methyl orange added (if the gas contains many particles, a bottle full of cotton wool must be placed

first to remove this. When the colour in the second bottle shows a slight tinge of redness the test must be stopped. The bottles are now taken off, well washed out with distilled water, and titrated back with the normal soda solution. Now 1 c.c. of the normal acid = 0.017 gram of ammonia.

Therefore, the number of c.c. of acid used multiplied by 0.017 gives the amount of ammonia in grams in the quantity of gas passed. This is easily calculated to grains per 100 cubic feet.

*Example:—*

Gas passed	10 cubic feet.
Acid taken	200 c.c.
Acid used	150 c.c.

Then  $150 \times 0.017 = 2.55 \times 10 \times 15.4 = 392.70$  grains of  $\text{NH}_3$  per 100 cubic feet.

**Carbonic Acid and Sulphuretted Hydrogen.**—This impurity and sulphuretted hydrogen are sometimes estimated together, but as the method consists of a series of U tubes containing calcium chloride, soda lime, and cupric phosphate, and it is at times very troublesome to get the gas in any appreciable quantity through these materials, I prefer to have two meters, and to take the gas from the same stream, separating by means of a T, and conducting one lot of gas through soda lime alone and the other through an acidulated solution of cadmium chloride. Both methods will be described:—

*Estimation of Carbonic Acid and Sulphuretted Hydrogen.*—The reagent employed for the estimation of the sulphuretted hydrogen is an impure di-tri-ortho-phosphate, the preparation of which is described in Chapter I.

The reagent is placed in a couple of U tubes, a small piece of cotton wool being placed in each stopper to prevent any of the reagent being mechanically carried forward by the gas.

The tubes are filled with this material and are then connected by rubber tubing together and about 3 cub. ft.

of clean coal-gas passed through them; this is rendered necessary because the inventor of this method, Mr L. T. Wright, F.C.S., found that the cupric phosphate gained in weight, but the increase soon reached saturation point, and he found the above-mentioned quantity of gas was sufficient for this purpose. The tubes are filled as follows:—They are first cleaned and dried, and one is filled with powdered calcium chloride; the stoppers well greased with vaseline or rubber grease; the next two U tubes are filled with cupric phosphate, great care being taken that the rough and fine portions are well mixed, otherwise the gas will have a too free passage through the cupric phosphate, and the  $\text{SH}_2$  will not be completely removed; the ground portions of the U tubes are now wiped free from dust, a little cotton wool placed therein, and the stopper well fitted in position. These are now ready for weighing after passing the usual quantity of clean coal-gas through them.

The carbon dioxide is absorbed by soda lime. It was found more expeditious to use the soda lime a little moist, as it absorbed more  $\text{CO}_2$  when moist. The necessary amount of moisture can be obtained by exposing the soda lime to the action of the air for twenty-four hours. These soda lime tubes remain very constant in weight; when clean, pure coal-gas is passed through them, and therefore it is not necessary to saturate them as in the case of the cupric phosphate. It is, however, necessary to pass pure dry coal-gas through them before weighing. A couple of U tubes are also filled with calcium chloride in the same manner as before. These are all weighed and are ready for passing the crude coal-gas through them.

The method of procedure is as follows:—

In cases (crude gas) where there is ammonia in the gas, this must also be removed, and is done as follows:—A U tube is filled with pieces of broken pumice which have been previously saturated with phosphoric acid.

As sulphuretted hydrogen is absorbed by vulcanised



and iron tubing it is necessary to have these thoroughly saturated with crude gas before starting the test.

In this test, as only a small quantity of gas is used, viz., about 0.5 cub. ft., it is necessary to have a blow-off cock, to keep the service fresh and clean, and also to have an average sample of gas.

The gas first passes through the phosphoric acid tube, and the outlet of this is connected to a large cylinder (similar to what is used for fouling new oxide of iron) filled with calcium chloride. The outlet of this drying cylinder is provided with a T-piece which is connected up with a Bunsen burner, which is kept burning during the test, keeping a good supply of fresh gas; the other arm is connected on to the soda lime tube, and the outlet of this on to the cupric phosphate tube or tubes, and next another calcium chloride tube, and then the soda lime tubes, and a calcium chloride tube last, and last the gas meter. The reason of these calcium chloride tubes is, the first one to make sure the gas is dried by the large cylinder, and the one after the cupric phosphate to absorb any moisture taken up by the dry gas from the cupric phosphate tubes, which must be added to the increase in weight of the cupric phosphate tube, and similar on the outlet of the soda lime tube. After the 0.5 cub. ft. has been passed, as shown by the meter register, which should occupy from one to one and a half hour, the tubes are disconnected, the stoppers being shut first. They are now wiped perfectly dry and are ready for weighing.

The increase in weight of each respective tube of material gives the direct amount of  $\text{SH}_2$  and  $\text{CO}_2$  in the quantity of gas taken, and can easily be calculated to grains per 100 cub. ft. The results so obtained will be a little high owing to the cyanogen in the gas being absorbed, but the error is slight and need not be considered.

*Example—*1. *Sulphuretted hydrogen :—*

Volume of gas passed, corrected to N.T.P. = 0.58 cub. ft.			
Weight of inlet calcium chloride tube	-	-	1168.9 grains.
"              "              "              "              after	-	-	1168.9 "
Increase - - -			<u>nil</u>
Weight of cupric phosphate tube A after	-	-	1200.9 grains.
"              "              "              A before	-	-	1196.1 "
SH <sub>2</sub> absorbed - - -			<u>4.8</u>

Tube B cupric phosphate showed no increase. The calcium chloride tube after cupric phosphate tube B likewise showed no increase; therefore 0.58 cub. ft. contains 4.8 grains of SH<sub>2</sub> = 827.59 grains per 100 cub. ft.

*Example—*2. *Carbon dioxide :—*

A. Weight of soda lime tube after	-	-	1468.8
"              "              "              before	-	-	1460.0
CO <sub>2</sub> absorbed - - -			<u>8.8</u>
B. Weight of soda lime tube after	-	-	1461.8
"              "              "              before	-	-	1461.6
CO <sub>2</sub> absorbed - - -			<u>0.2</u>
Calcium chloride tube after	-	-	1261.3
"              "              before	-	-	1261.0
Increase - - -			<u>0.3</u>

The increase in the calcium chloride tube is due to the dry gas absorbing moisture from the moist soda lime, and must be added to the increase of the soda lime; therefore

$$8.8 + .2 + .3 = 9.3 \text{ increase due to CO}_2;$$

therefore there is 9.3 grains CO<sub>2</sub> in 0.58 cub. ft. = 1603.44 grains CO<sub>2</sub> per 100 cubic feet.

It will be obvious that there is a certain amount of error which is multiplied by a very large factor in taking such a small quantity of gas. The next method described will be a separate test for  $\text{CO}_2$  and  $\text{SH}_2$ , but the quantity of gas passed can be as much as is desired, and the test can be put on and left for a period of twenty-four hours, giving a much better average and a far more stringent test.

*Estimation of Carbon Dioxide in Coal-Gas or Oil Gas.*

Three large cylinders are filled with soda lime, and weighed on an accurate balance, after being blown with pure coal gas. A couple of U tubes are filled with calcium chloride as usual.

The apparatus is connected up as follows: The gas is passed through an oxide purifier to free it of  $\text{SH}_2$ , then through a meter, then through a large cylinder of calcium chloride, and next through one of the small U tubes filled with calcium chloride, the outlet of which is connected up to inlet of No. 1 of the soda lime cylinder, which is in turn connected to No. 2 and No. 3 soda lime cylinders, the other calcium chloride tube coming last, the outlet of which is connected on to a burner, and the gas is burnt. The inlet also has a blow-away cock which burns the gas by means of a burner, so keeping the service fresh.

The quantity of gas passed can be regulated to any amount during the day, but I have found 10 to 12 cub. ft. sufficient. The tubes are disconnected and weighed as usual, the increase in weight giving the amount of  $\text{CO}_2$  in the quantity of gas taken; this multiplied by 15.439 = grains, and can be calculated to grains per 100 cub. ft. If it is desired to express the result in percentage by volume, proceed as follows: A cubic foot of dry carbonic acid weighs 817.3 grains, therefore dividing the number of grains per 100 cub. ft. by 817.3 equals the percentage by volume of  $\text{CO}_2$  present in the gas.

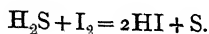
*Estimation of Sulphuretted Hydrogen by Cadmium Chloride.*—A saturated solution of cadmium chloride, which has been slightly acidulated with hydrochloric acid is used

for this test. 500 c.c. of this solution are placed in three Woulfe bottles, say 200 c.c. in first and second, and 100 c.c. in the third.

Gas is now passed through these bottles, which are connected up as usual, and the presence of  $\text{SH}_2$  is noted by the solution giving a yellow precipitate. When this precipitate arrives at the second Woulfe bottle, attention must be given, and directly the third bottle turns the slightest yellow, the test must be shut off.

There is a blow-off cock as in similar cases to get a good, fresh, and average supply of gas. The gas is passed at the rate of about 0.5 cub. ft. per hour, and must not be allowed to bubble too quickly through the cadmium chloride. The bottles are now washed out with a little distilled water, to which has been added a few drops of hydrochloric acid; the whole is now placed in a beaker, and an excess of bromine water added, or neat bromine added, to oxidise the cadmium sulphide to a sulphate. When completely oxidised, and the excess of bromine boiled off, add hydrochloric acid, and precipitate the sulphur by addition of barium chloride, which will give a white precipitate of barium sulphate. This is filtered, and well washed till free from chlorides, and incinerated in a platinum crucible and weighed as  $\text{BaSO}_4$ . The weight of  $\text{BaSO}_4$  multiplied by .1459 gives the weight of sulphuretted hydrogen in the quantity of gas taken.

Another method for estimation of  $\text{SH}_2$  is as follows:— A standard solution of iodine is made up so as to be of a decinormal strength, the titration being effected by a decinormal sodium hyposulphite solution, starch being used as indicator. The following reactions take place:—



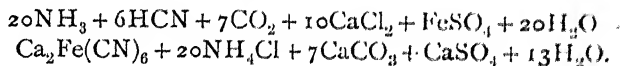
The test is carried out with a Wanklyn bottle (which holds  $\frac{1}{10}$  of a cub. ft.). The results at the best are only very approximate, because undoubtedly the sulphur compounds other than sulphuretted hydrogen have an effect on the test, causing an incalculable error.

**Carbon Disulphide.**—This compound is estimated by what is called the Referee sulphur test, and is given under that heading.

**Cyanogen.**—There are many methods on the market for extracting the cyanogen from coal-gas. The cyanogen exists in the gas in the form of hydrocyanic acid gas, and there is a great deal of difference of opinion as to the best method of arresting this important compound.

In some works it is arrested directly after the exhausters or condensers by a solution of ammonium ferrosulphide solution; this is made by adding a certain quantity of "copperas" or iron sulphide to a certain strength solution of ammonia (which is obtained by washing the gas with water), forming a solution of ammonium ferrosulphide solution, which, when the gas containing the hydrocyanic acid gas is washed by this solution, forms ammonium ferrocyanide.

There are numerous other methods for extraction of the cyanogen from gas. Feld proposes to extract it by using calcium chloride and ferrous sulphate. The following equation expresses the result :—



Dr J. Crossman, in December 1903, read a paper before the Society of Chemical Industry. The reactions upon which he bases his process are briefly summed up in the following equations :—

- (1.)  $2\text{Na}_4\text{FeCy}_6 + (3 + x)\text{H}_2\text{SO}_4 = 6\text{HCy} + \text{Na}_2\text{Fe}_2\text{Cy}_6 + 3\text{Na}_2\text{SO}_4 + x\text{H}_2\text{SO}_4$
- (2.)  $3\text{Na}_2\text{Fe}_2\text{Cy}_6 + 6\text{NaOH} + 4\text{O} = 3\text{Na}_4\text{Fe}_2\text{Cy}_6 + \text{Fe}_3\text{O}(3 + 4) + 3\text{H}_2\text{O}.$
- (3.)  $\text{NaOH} + \text{HCy} = \text{NaCy} + \text{H}_2\text{O}.$

As seen, the Cy is extracted by soda, and afterwards decomposes with  $\text{H}_2\text{SO}_4$ , &c., making either sodium cyanide or hydrocyanic acid liquid.

The other method of arresting the hydrocyanic acid gas is to allow as much as will go forward to the purifiers, where it enters into combination with the oxide of iron, forming a cyanide of iron. The method for extracting this from the spent oxide will be given when dealing with spent oxide. The drawback in this latter method is that a certain portion of the total HCN in the gas is lost by washing with the liquid in the extraction of ammonia, forming ammonium sulphocyanide, &c., which do not pay to extract the amount of cyanogen.

In the analytical experiment for the estimation of the total hydrocyanic acid the method is as follows:—A 30 per cent. solution of caustic potash is made up by dissolving 300 grams of caustic potash in a litre of water, and a 10 per cent. solution of sulphate of iron is prepared in a similar manner. These two solutions are now mixed in the proportion of 4 of the potash to 1 of the iron. This is now placed in four small Woulfe bottles, and the gas passed through these, forming ferrocyanide of potassium. The test is stopped when the last bottle begins to turn blackish. The meter is read to know the quantity of gas passed. The bottles are washed out and mixed, and a certain portion taken and boiled, still free of ammonia; it is then filtered and the precipitate well washed.

This solution contains certain impurities, the chief one being sulphur, which comes down with the Prussian blue, causing a considerable error. To eliminate these impurities acidify with hydrochloric acid, and add barium chloride, which precipitates the sulphur as barium sulphate, filter, and precipitate the cyanogen as Prussian blue, by adding an excess of ferric chloride, allow to stand for a little while on water bath, and then filter on a tared filter paper, wash well, dry in oven and weigh; calculate the weight on the total quantity of caustic potash and iron solution used gives the amount of Prussian blue in the quantity of gas taken; this is easily calculated to lbs. of Prussian blue per ton of coal carbonised.

In the estimation of Prussian blue care must be taken not to take too large a proportion of the solution as, if the precipitated blue is very heavy, it is very difficult to wash it free of excess of iron.

The amount of Prussian blue found can easily be calculated to grains of hydrocyanic acid per 100 cub. ft.

This is far the best way of expressing the result, as it is shown how the cyanogen exists in the gas.

*Example—*

Gas used on experiment = 134.5 cub. ft.

Amount of Prussian blue = 22.40 grams.

Make of gas per ton of coal carbonised = 10,700 cub. ft.

Therefore  $22.40 \times 10,700 \div 134.5 = 1782.74$  grains of Prussian blue per ton of coal.

Thence  $1782.74 \div 453.6 = 3.93$  lbs. Prussian blue per ton of coal carbonised.

To express the lbs. of Prussian blue in grains of hydrocyanic acid per 100 cub. ft. proceed as follows:—

$$1782.74 \times 15.4 = 27454.2 \text{ grains.}$$

$$27454.2 \times 468 = 12848565.6 \text{ grains.}$$

$$12848565.6 \div 860 = 14940.19 \quad "$$

$$14940.19 \times 27 \div 26 = 155.32 \text{ grains HCN.}$$

Therefore 155.32 grains of hydrocyanic acid per 100 cub. ft., which is equivalent to 3.93 lbs. Prussian blue per ton of coal carbonised.

The amount of Prussian blue can be estimated volumetrically by a standard solution of zinc sulphate made by dissolving 45 grams of pure zinc sulphate in a litre of water. This solution is standardised by titration against a standard solution of potassium ferrocyanide, 5 per cent.

1 c.c. of the  $\text{ZnSO}_4 = 1$  c.c. potassium ferrocyanide.

1 c.c. " " = 0.05 gram potassium ferrocyanide.

Multiply the numbers of c.c. of zinc sulphate solution used by 0.687 = Prussian blue,  $\text{Fe}_4\text{Fe}_3\text{Cy}_{18}$ . The indicator in this case is ferric chloride, and is used on the spot reaction paper as in the estimation of iron in bog-ore.

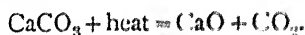
## CHAPTER VI.

### ANALYSIS OF LIME.

THE analysis of lime from a gas chemist's point of view means the estimation of the total amount of free lime or caustic lime (CaO).

The lime used in a gasworks is what is known as "flare" lime. This is prepared from the purest chalk, and the term "flare" is derived from the method employed in burning the chalk to lime.

The flare from a furnace burns the chalk to lime as per the following equation :—



The old-fashioned kiln method in which the fuel and chalk are mixed together, or else laid in a layer of fuel and then a layer of chalk, gives a similar result, but requires "picking" afterwards to get rid of the clinker, and the lime formed is not so pure, as the ash of the fuel forms various calcium silicates.

In the analysis of chalk, all that is required is the estimation of the carbon dioxide, which theoretically is 44 per cent. of  $\text{CO}_2$ , from the equation  $\text{CaCO}_3 = 100$ ,  $\text{CaO} = 66$ , and  $\text{CO}_2 = 44$ . In the analysis of "flare" lime the carbon dioxide ( $\text{CO}_2$ ) and the caustic lime (CaO) are estimated.

There are numerous methods for carrying these out on a works where it is only necessary to get "approximate" results to show how a kiln is working, and it is only required to estimate the amount of carbon dioxide to find how the plant is going.



The quickest and easiest method is to estimate the  $\text{CO}_2$  by some sort of calcimeter, or an apparatus known as the Schrötter apparatus. The figure below shows the Scheibler's calcimeter. The working of the apparatus is as follows:—A known quantity of lime is taken (say .5 gram) and is put into bottle A, tube B in bottle A being filled with a 33 per cent. solution of hydrochloric acid. The stopper is then closed, and the bottle turned on one side, so that the hydrochloric acid flows out on to the lime, the carbonic acid is then evolved and passes over into the bag C, which is inflated with the gas and acts on the air in the bottle, and so sends the water down the graduated arm of the burette with a corresponding increase in the other arm. The amount of decrease in the water is read off, and, multiplied, gives the percentage of  $\text{CO}_2$  in the sample. This result is only approximate, but very good results can be obtained if care is taken in keeping the apparatus in the same place, and the temperature about the same day by day. Of course this apparatus is not suitable for small percentages of  $\text{CO}_2$ , but anything above 5 per cent. gives very fair results.

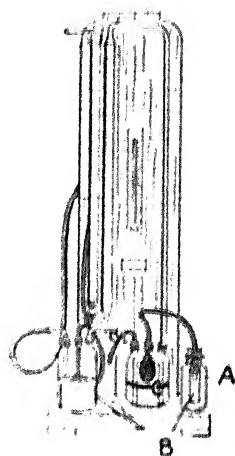


FIG. 30.  
SCHEIBLER'S CALCIMETER.

In the use of the Schrötter apparatus the result is estimated by difference, *i.e.*, a known weight of lime or chalk is taken, the  $\text{CO}_2$  evolved, and the flask and contents weighed again. Decrease in weight is  $\text{CO}_2$ .

Fig. 31 shows the Schrötter apparatus. A known quantity of lime or chalk is weighed out and put into the flask at the stopper on side of flask marked A. The acid is admitted from the stoppered funnel C, while the escaping carbon dioxide is dried by its passage through the strong sulphuric acid contained in B. The gas passes up

the central tube within B, and forces the acid down to the level of the holes near the bottom of the outer tube, and then bubbles out through the acid and escapes at the top of the outer tube.

Neither of these apparatus are suitable for the estimation of  $\text{CO}_2$  in lime or chalk, if these substances contain any volatile matter that is evolved on the addition of hydrochloric acid, such as sulphuretted hydrogen, which is present in reburnt lime.

The method to employ for accurate determination of carbon dioxide is by absorbing the  $\text{CO}_2$  either in a Geissler's & Mohr potash bulb, or in a U tube filled with soda lime.

The method of procedure is as follows:—

Fig. 32 represents the apparatus used for the process.

The method of working is as follows:—1 or 2 grams of lime or chalk are taken and put into flask A; a solution of hydrochloric acid (33 per cent.) is added through safety funnel; the carbonic acid gas evolved is driven through

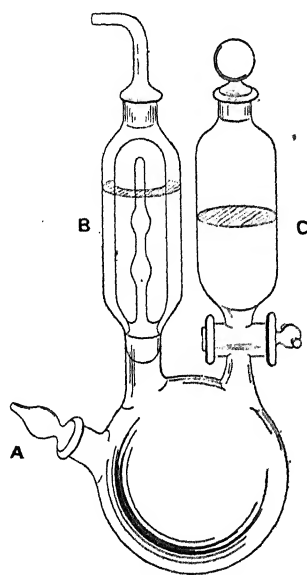


FIG. 31.—SCHRÖTTER APPARATUS.

the U tubes and is absorbed in G. The cupric phosphate tubes are only necessary when testing spent or reburnt lime, to retain the sulphuretted hydrogen driven off. The calcium chloride tubes are weighed before and after the experiment, together with the Geissler bulb and drying tube; the increase in weight of the latter giving the amount of  $\text{CO}_2$  in the quantity taken, which can be easily related to percentages.

After describing the various methods for the estimation of carbon dioxide, the next estimation is for total lime.

The usual quantitative method is to dissolve a weighed quantity, say 50 grams, in hydrochloric acid, and take an aliquot part of this and add alcohol (spirit of wine) in the

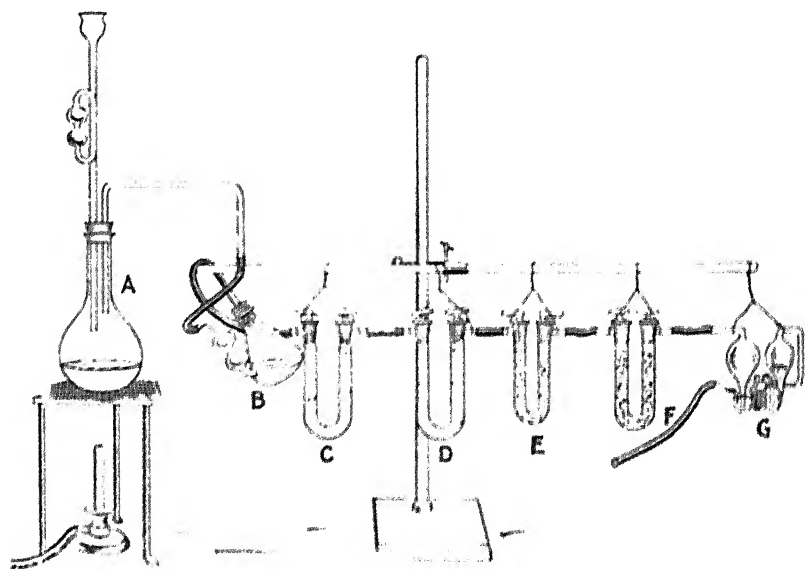


FIG. 32.—APPARATUS FOR DETERMINING THE  $\text{CO}_2$ .

*A* is the evolution flask; *B*, bulb containing sulphuric acid; *C*, U tube containing cupric phosphate; *D*, ditto; *E* and *F*, U tubes containing calcium chloride; and *G*, Geissler's bulb, with calcium chloride tube attached.

proportion of about two-thirds of the spirit to one-third of the solution. After thoroughly mixing add sulphuric acid in a slight excess.

Calcium sulphate ( $\text{CaSO}_4$ ) is precipitated. The solution is then filtered, and the filter paper washed with dilute spirit, and eventually dried, ignited, and weighed.

The calculation is then very easy from the following factors :—

1 grain $\text{CaSO}_4$	= 0.412 CaO.
1 " $\text{CO}_2$	= 1.272 CaO.
1 " $\text{CO}_2$	= 2.272 $\text{CaCO}_3$ .

Mr H. Leicester Grevelle, F.I.C., F.C.S., gives a most useful method (*Journal Gas Lighting*, 17th October 1905). It is founded on the fact that free lime, though having a limited solubility in water, is very much more soluble in strong solution of pure cane sugar or glycerine.

His experiments were made on sugar solution. These were placed in a 20 oz. flask, 100 grams of the finely ground sample, and distilled water added to effect slaking. The sugar solution was then added and the mass digested for about half an hour at a gentle heat, the solution diluted and made up to a measured volume, and a portion titrated with standard sulphuric acid with any suitable indicator. He obtained concordant results with the  $\text{CaSO}_4$  method. The advantage claimed is that it is volumetric and only takes the free lime into account, rendering a special determination of the proportion of carbonate unnecessary. The amount of CaO in a good sample of flare lime should not be less than 94.0 per cent., and the  $\text{CO}_2$  not higher than 3.0 per cent.

Another method ought to be mentioned here that has been tried with varying results. The method is as follows :—A weighed quantity of lime is slaked, dried in a water bath at 212 degrees Fahr., and weighed again. The increase in weight is water absorbed to form calcium hydroxide,  $\text{Ca(OH)}_2$ . The method is as follows :—The sample of lime is well mixed and 10 grams are weighed out in a porcelain dish. Water is then added in excess to thoroughly slake the lime.

The basin and contents are now placed in a water oven and dried until weight is constant. The increase in weight is the amount of water absorbed by the lime ( $\text{CaO}$ ) to

form the hydrate  $\text{CaH}_2\text{O}_6$ , as  $\text{CaO} + \text{H}_2\text{O} = \text{CaH}_2\text{O}_6$ . Now the water in the hydrated lime is not decomposed at 100 degrees Cent., therefore the increase in weight gives the amount of water absorbed to form the hydrate. Now 18 parts of water absorbed represent 56 parts of caustic lime. In this the increase in weight was 3.1, which equals 31.0 per cent.

Therefore  $18 : 31 :: 56 : 95.88$ ,  
which equals 95.88 per cent. caustic lime.

The best gravimetric method for estimating the calcium oxide ( $\text{CaO}$ ) in a sample of lime is as follows :

Weigh out 1 gram of the sample and dissolve it in a weak solution of hydrochloric acid, pouring the acid gently down the side of the beaker till all effervescence ceases. When it has all been dissolved that will dissolve, filter, to separate the undissolved matter (silica, &c.).

The beaker should have a watch glass placed over it during the time it is dissolving, as on the addition of the acid it is liable to splash. The watch glass is then washed into the beaker, and to the filtrate ammonia is added until the solution smells strong of the reagent. The liquid is now heated to boiling. The calcium in the form of calcium oxalate is now precipitated by the addition of a slight excess of a warm saturated solution of ammonium oxalate, to which a little ammonia has been added. The beaker and contents are kept at the boil for a few minutes and then allowed to settle. The clear liquid is now decanted off through a filter without disturbing the precipitate. The precipitate is now washed three or four times in the beaker, by addition of distilled boiling water, and allowed to settle each time, and the washing poured on to the filter. The small quantity of precipitate poured on the filter paper at each washing will so far fill up the pores of the paper that when, after the third or fourth washing, the precipitate is finally poured on the filter paper the filtrate will come through perfectly clear. The precipitate is then washed with hot water until the filtrate is free from chlorides.

indicated by the absence of any milkiness on the addition of a few drops of silver nitrate acidifying with nitric acid. The filter paper and contents are now put into the water bath, and when dry the precipitate is transferred as completely as possible into a platinum crucible, the filter paper being incinerated separately, and the ash added to the precipitate in the crucible.

The precipitate (calcium oxalate) is then converted into the carbonate by a gentle heat, care being taken that the crucible never reaches a visible redness at the bottom of the crucible. After gently heating for about twenty minutes, cool in desiccator and weigh. This gives the calcium oxide in the sample in the form of carbonate. The calcium carbonate can now be converted into calcium oxide by further heating the crucible for about ten minutes to a red heat, and finishing for a few minutes with a blow-pipe flame. Cool as before, and again weigh; the operation is repeated until there is no further loss in weight. This gives the calcium oxide in the sample.

**The Estimation of Silica and Alumina.**—The residue remaining after the evolution of carbonic acid, and which has not been dissolved by hydrochloric acid, is sand and clay. A small quantity of what is called soluble silica will be found in the hydrochloric acid solution. No difference is generally made in the estimation of silica, whether soluble or insoluble, but the total is estimated as follows :—

The total contents of the flask are rinsed out into a porcelain dish, a little strong nitric acid added, and the liquor evaporated to dryness. The porcelain dish is then gently heated on a sand bath until all moisture is driven off, which is ascertained by holding a watch glass over porcelain dish, when no moisture should be deposited.

The porcelain dish is now allowed to cool, and the contents are heated with a little strong hydrochloric acid, everything except the silica is dissolved. A little

water is now added, and the precipitate filtered off, the precipitate being well washed with water until free from chloride (ascertained by adding a drop of silver nitrate to a little of the filtrate in a test-tube, when it should remain clear, a slight cloudiness showing presence of chlorides).

The residue is now dried in the water oven (paper and all) and next ignited in a platinum crucible. The residue is generally more conveniently incinerated apart from the filter paper, the filter paper being added to crucible afterwards. The weight of residue obtained equals silica in the amount taken, and can easily be calculated to percentage. The filtrate contains the iron and alumina, which are in the form of their respective oxides, viz., ferric oxide,  $\text{Fe}_2\text{O}_3$ , and aluminium oxide,  $\text{Al}_2\text{O}_3$ .

These are estimated separately.

**Separation of Iron and Alumina.**—A considerable quantity of ammonium chloride is added to the filtrate and gently warmed; ammonia is now added in slight excess, and the mixture boiled; this precipitates both the iron and alumina hydroxides. They are now filtered and washed. The precipitate is dissolved in the filter by pouring a little warm dilute hydrochloric acid, and the solution poured into a strong solution of potassium hydroxide (free from alumina) contained in a platinum dish, and the mixture is then boiled for two or three minutes. The iron is precipitated as ferric hydroxide, the alumina remaining in solution as potassium aluminate. The precipitated ferric hydroxide is now filtered off, well washed with water, then re-dissolved in hydrochloric acid and re-precipitated by slight excess of ammonia. The ferric hydroxide is again filtered, washed, and dried in the usual way, and ignited and weighed in the form of  $\text{Fe}_2\text{O}_3$ . The total filtrates are acidified with strong hydrochloric acid, and the aluminium precipitated as hydroxide by addition of a slight excess of ammonia. The precipitate is washed and dried and ignited and weighed as  $\text{Al}_2\text{O}_3$ .

Sometimes the material contains manganese, which however is in very small quantities, and is not usually analysed separately for.

The analysis of spent lime is very difficult, as the various sulphides formed are continually undergoing oxidisation, thereby altering their original state. It is very rare indeed to analyse a sample of spent lime, except for the amount of carbonate and sometimes for the amount of free lime that is left unconverted in carbonate.

For estimation of carbonate proceed as in chalk or flare lime, taking care to keep back any sulphuretted hydrogen.

For estimation of free lime, or rather hydrated as  $\text{Ca}(\text{HO})_2$ , proceed as follows:—The process consists in adding an excess of a solution of a copper salt to a weighed quantity of lime. The  $\text{CaO}$ ,  $\text{H}_2\text{O}$  gives a precipitate of  $\text{CuO}$ ,  $\text{H}_2\text{O}$ , the excess of the  $\text{Cu}$  remaining unaltered in the solution. Knowing how much  $\text{Cu}$  salt was first added, and then determining the amount remaining in solution, the difference between the two represents the amount of  $\text{Cu}$  thrown out of solution as hydrate. The equivalent of this quantity of  $\text{CuO}$ ,  $\text{H}_2\text{O}$  in terms of  $\text{CaO}$ ,  $\text{H}_2\text{O}$  is the amount of  $\text{CaO}$ ,  $\text{H}_2\text{O}$  in the quantity operated on. A weighed quantity of spent lime is weighed out (say 10 grams) and is placed in a 100 c.c. measure, and standard cupric chloride solution added up to the measuring line. The mixture is allowed to stand a few hours, and is periodically shaken up. A measured quantity (say 20 c.c.) of the clear liquid is taken out by means of a pipette, placed in a suitable vessel, and an excess of  $\text{NH}_4\text{HO}$  added—the blue liquid formed diluted to a convenient strength with distilled  $\text{H}_2\text{O}$ .

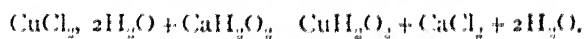
$\text{NH}_4\text{OH}$  and distilled water are next placed in a similar vessel, and some of the standard cupric chloride solution run in, until the same depth of colour is obtained by looking down the liquid on to a white surface.

Ascertaining how much of this standard cupric chloride solution is equal to that present in the 20 c.c. of liquid



taken, it is easy to calculate the amount equal to that in the whole 100 c.c. The amount of cupric chloride present in the original 100 c.c. being known, and that now present being ascertained, the difference between the two quantities is the amount of CuCl<sub>2</sub> removed as hydrated from the solution. Its equivalent of CaO, H<sub>2</sub>O can therefore be calculated.

The reaction is represented by the formula : —



## CHAPTER VII.

### *AMMONIA.*

THE great bulk of ammonia and ammonia compounds is obtained from gasworks, where decided efforts are made to remove all the ammonia that exists in the gas by washing with water, which absorbs the ammonia.

In the Forty-Second Annual Report on Alkali, &c., Works, by the chief inspector, published in July 1906, the following Table is given :—

RECOVERY AND PRODUCTION OF AMMONIA.  
AMOUNT OF SULPHATE OF AMMONIA PRODUCED IN THE  
UNITED KINGDOM (TONS).

	1905.	1904.	1903.
Gasworks - - - - -	155,957	150,208	149,489
Ironworks - - - - -	20,376	19,568	19,119
Shaleworks - - - - -	46,344	42,486	37,353
Coke oven works - - - - -	30,732	20,848	17,438
Producer gas and carbonising works (bone and coal) -	15,705	12,880	10,265
Total - - - - -	269,114	245,990	233,664

The most important contributor still of course remains the gas industry.

The next subheads following gasworks in the Table the produce from coal used in blast furnace opera-

tions in ironworks, and from shale used in the production of paraffin oil. The other items explain themselves.

The total nitrogen in coal varies from 1 to 2 per cent., but in the destructive distillation of coal nothing like the whole of the nitrogen escapes in the form of ammonia. (Lunge, "Coal-Tar and Ammonia.")

As early as 1863 A. W. Hofmann stated that coal in carbonising only yields one-third of its nitrogen, two-thirds remaining in the coke. Dr Tidy remarks that if all the nitrogen in coal reappeared as ammonia in the gas liquor, it would yield per ton of coal from 142 to 226 gallons of liquor of 4 degrees Tw., while in practice rarely more than 45 gallons is obtained.

W. Foster (*Jour. Chem. Soc.*, xliii, p. 105) showed that, of 100 parts of nitrogen contained in coal, there were obtained in a laboratory experiment :—

14.50	parts as ammonia,
1.50	" " cyanogen,
35.26	" in the elementary condition (as part of coal gas),
48.68	" remaining in the coke.

Watson Smith (*Jour. Soc. Chem. Ind.*, 1883, p. 438) found that coal-tar, which Foster neglected in his calculations, contained 1.667 per cent. N (pitch containing 1.595, and coal-tar oil about 2 per cent.), that is, not quite 0.1 per cent. N calculated upon the coal from which the tar is derived. In coke he found :—

Ordinary gas coke	1.375	per cent. nitrogen.
Bee hive coke	0.511	" "
Coke from Simon Carres ovens	0.384	" "

This shows that much less nitrogen is driven out of coal in the short process of gasmaking than in the long-continued process in the manufacture of metallurgical coke. There are many methods for increasing the yield of ammonia from coal, such as adding lime, treating coal

with steam. For further information of this matter the reader is referred to Lunge, "Coal-Tar and Ammonia."

Ordinary gas liquor can be divided into two classes, viz., the volatile ammonia, and the fixed ammonia. The fixed ammonia is formed during the scrubbing or washing of the gas, when the ammonia enters into chemical combination with the carbon dioxide, sulphur, and cyanogen. These substances are :—

I. *Volatile at ordinary Temperatures—*

- Ammonium carbonate (mono, sesqui, bi).
- „ sulphite,  $(\text{NH}_4)_2\text{S}$ .
- „ hydrosulphide,  $\text{NH}_4\text{HS}$ .
- „ cyanide.
- „ acetate (?).
- Free ammonia.

(The presence of free ammonia is doubted by most chemists.)

II. *Fixed at ordinary Temperatures—*

- Ammonium sulphate.
- „ sulphite.
- „ thiosulphate (hyposulphite).
- „ thiocarbonate.
- „ chloride.
- „ thiocyanate (sulphocyanide).
- „ ferrocyanide.

— Lunge.

The Alkali Report for 1905, pages 35 and 36, gives the following analysis of samples of English gas liquor :—

# ANALYSIS OF GASWORKS' LIQUOR.

[illegible]

**The Analysis of Ammoniacal Gas Liquor.**—This is not unfrequently carried out in a gasworks by means of a hydrometer, but generally only as a very rough guide.

Twaddell's hydrometer is generally used for this purpose, and the rule is:—Degrees Twaddell multiplied by 2 equal oz. of ammonia in liquor per gallon.

But this process, which is really the specific gravity of the liquor, is most deceptive, and is certainly in the favour of the buyer and against the gas company, for the ammonium salts raise the density of the solution in an unequal degree, and free ammonia (which seldom occurs) lowers it.

This is proved by the following experiment, the liquor being tested by Twaddell hydrometer, then distilled (as explained later) for the total ammonia, both free and fixed:—

Degrees on Twaddell hydrometer =  $3 \times 2 = 6.00$  oz.

By Will's distillation test = 7.80 oz.

The following Table, given in Lunge's "Coal-Tar and Ammonia," still further proves the point:—



FIG. 33.  
TWADDELL'S  
HYDROMETER.

Degrees Baumé	2°	2.5°	3°	3.5°	4°	4.5°	5°	6°
Specific Gravity	1.0138	1.0163	1.0208	1.0249	1.0280	1.0316	1.0352	1.0426
Per cent. $\text{NH}_3$	1.16	1.30	...	...	...	...	...	...
" "	1.42	1.43	...	...	...	...	...	...
" "	1.50	1.63	1.63	...	...	...	...	...
" "	1.77	1.77	1.76	1.87	...	...	...	...
" "	...	1.98	1.90	2.00	...	...	...	...
" "	...	2.18	2.10	2.24	...	...	...	...
" "	...	2.65	2.38	2.40	2.55	...	...	...
" "	...	...	2.45	2.72	2.72	2.79	...	...
" "	...	...	...	...	2.90	2.85	...	...
" "	...	...	...	...	3.40	3.06	...	...
" "	...	...	...	...	...	3.40	...	...
" "	...	...	...	...	...	3.53	3.67	3.74

These tests, which have been proved correct by T. H. Davis (*Chemical News*, xxxviii., p. 193) and others, show that it is decidedly wrong to value a gas liquor by its gravity. It is therefore decidedly preferable to value gas liquor by a chemical test.

There are two methods in use: the general one in use in England is to state the amount of ammonia in oz. per gallon. This refers to the number of ozs. of real sulphuric acid ( $\text{H}_2\text{SO}_4$ ) required to saturate or neutralise 1 gallon of liquor.

The standard acid is made by diluting 16.5 oz. of  $\text{H}_2\text{SO}_4$  with water, and making up the total to 1 gallon. The specific gravity of this standard solution at various temperatures is as follows:—

Temperature.	Specific Gravity.	Temperature.	Specific Gravity.	Temperature.	Specific Gravity.
Degrees Fahr.		Degrees Fahr.		Degrees Fahr.	
50	1.06640	57	1.06503	64	1.06356
51	1.06621	58	1.06482	65	1.06335
52	1.06602	59	1.06461	66	1.06314
53	1.06583	60	1.06440	67	1.06293
54	1.06564	61	1.06419	68	1.06272
55	1.06545	62	1.06398	69	1.06251
56	1.06524	63	1.06377	70	1.06230

When the acid solution is as near as possible to the desired gravity, it must be tested either by titrating against sodium carbonate, or 10 c.c. may be precipitated with barium chloride, when it should yield 2.378 grams of barium sulphate. This solution is then run into 16 c.c. of the gas liquor to be tested, until it is exactly neutral, as shown by litmus paper, or methyl orange, or some suitable indicator. The number of c.c. of standard acid used gives the ozs. of free ammonia and ammonium salts per gallon; this is generally called the acid test or saturation test, but is no indication of the real value of a sample of gas liquor.

When the total ammonia, including both free and fixed,

is required to be estimated, a known quantity of liquor is distilled with a solution of potash or soda in excess into a known quantity of standard acid. It is necessary to prepare a standard sodium hydrate solution to exactly equal the standard acid, *i.e.*, 1 c.c. of the standard acid will be exactly neutralised by 1 c.c. of the standard soda solution. Therefore as each c.c. of the standard acid used to neutralise the liquor equals 1 oz. of ammonia per gallon, the soda solution will therefore be of equal strength.

The method of procedure is as follows:—16 c.c. of the gas liquor to be tested are placed in the flask C, excess of a strong solution of potash is added through funnel E, stop-cock closed, 16 c.c. of the standard acid are placed in flask G, and sufficient distilled water, so that the tube F will be submerged. The gas is now lighted, and the gas liquor kept boiling steadily for an hour or more, until all the ammonia is driven off, proved by opening cock E, and holding a turmeric paper there, which instantly turns red if any ammonia is present.

On completion of distillation the flask G is washed out and titrated by the standard soda solution to ascertain how many c.c. of the acid solution have been neutralised by the ammonia in the gas liquor.

The number of c.c. of soda solution used, deducted from 16 c.c. (the number of c.c. of acid used), gives the number of oz. per gallon of liquor.

*Example—*

16 c.c. of acid = 16 c.c. of soda.

16 c.c. of acid used for distillation; 7.8 c.c. of soda used to neutralise acid solution after distilling the gas liquor.

Therefore 16 c.c. - 7.8 c.c. = 8.20 oz. of ammonia per gallon.

This is what is known as Will's distillation test. (The other method for estimation of the percentage of ammonia is described under ammonium sulphate.) These are the usual tests carried out on a gasworks, but it is sometimes necessary to have a complete analysis of a gas liquor.



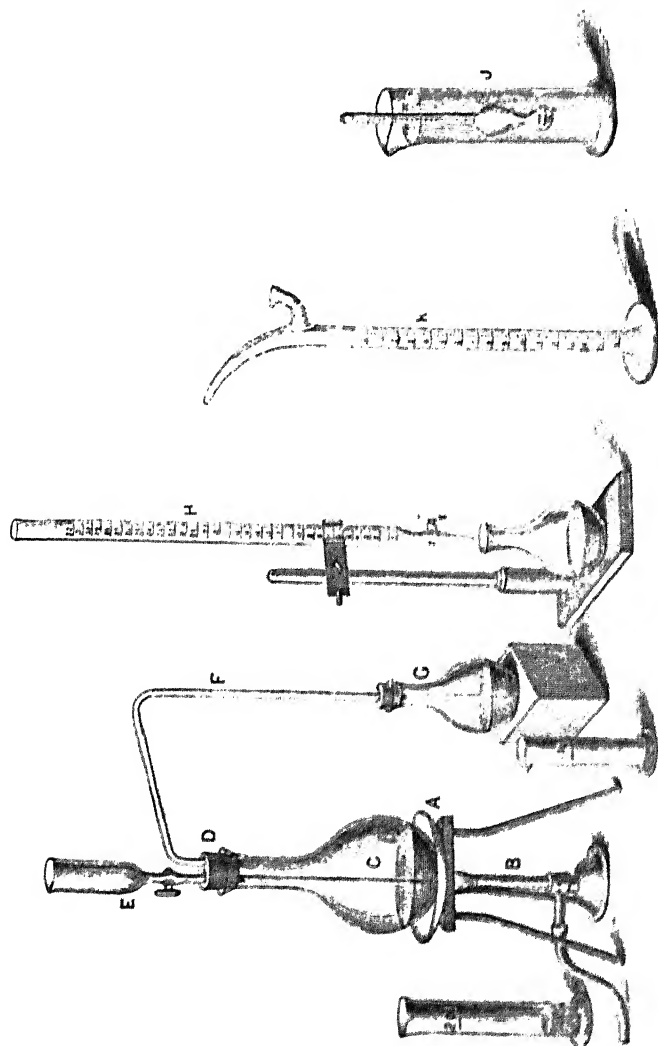


FIG. 14.—APPARATUS FOR THE ESTIMATION OF AMMONIA.  
*A.* Small flask; *B.* Round-bottom flask; *C.* Thermometer; *D.* Gas-bottle; *E.* Gas-washing bottle; *F.* Gasometer; *G.* Gas-bottle; *H.* Large graduated cylinder; *I.* Small flask.

The foregoing tests are from the Report of the Chief Inspector of Alkali Works. The complete report on analyses of ammoniacal liquor from various sources is contained in the Fortieth Annual Report, and one or two additions have been made in some of the subsequent ones.

### Ammonia.

1. **Free Ammonia**.—(A) *By Direct Titration* (to determine approximately the volume of acid required for distillation (B)).—10 c.c. of liquor are diluted to 100 c.c. and titrated with  $\frac{N}{2}\text{H}_2\text{SO}_4$ —methyl orange indicator.

(B) *By Distillation*.—10 c.c. liquor (more if weak) diluted to about 300 c.c. in round-bottomed flask connected through a catch bulb to Liebig's condenser and receiver, containing excess of  $\frac{N}{2}\text{H}_2\text{SO}_4$  and provided with outlet acid catch packed with broken Jena glass (some beads are found to yield alkali to  $\frac{N}{2}$  acid, and their use is not recommended); at the close of distillation air is blown through the apparatus to remove final traces of ammonia.

150 c.c. of the solution are distilled, the excess of acid in receiver is titrated with  $\frac{N}{2}\text{Na}_2\text{CO}_3$ , then 100 c.c. further distilled into receiver without acid, titrated with  $\frac{N}{2}\text{H}_2\text{SO}_4$ .

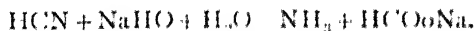
The amount of ammonia obtained by this second distillation is in general nil. In exceptional cases, however, e.g., producer gas liquors, evolution of ammonia from decomposition of nitrogenous organic matter was so continuous as to make it impossible to complete the distillation for "free" ammonia.

In such cases it was only possible to present a figure for "total" ammonia (free and fixed), addition of caustic soda by effecting the decomposition of ammonia-yielding compounds.

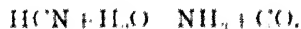
**Fixed Ammonia**—(C) *By Distillation.* Add boiled caustic solution in excess, with sufficient water to replace that distilled off, and proceed as above.

The whole of the ammonia is found in the first 150 c.c. of distillate, with rare exceptions. In such exceptional cases the distillation is prolonged with addition of more water.

(Attention is directed to the behaviour of cyanides on distilling a gas liquor containing these with caustic soda. It was proved that hydrocyanic acid when boiled with caustic soda yielded sodium formate and ammonia, as per the equation :—



It was also found that cyanogen compounds were decomposed by steam resulting from oxidation of the sulphuretted hydrogen by air :—



A reaction analogous to the formation of ammonia from metallic cyanides when heated with steam.)

*Calculations*—

$\text{NH}_3$  grams per 100 c.c. of liquor  $.0085 \times 10 \times \text{c.c.} \frac{\text{N}}{2}$  acid.

H.E. (Hydrogen Equivalent)  $\frac{\text{Free } \text{NH}_3 \text{ grams}}{.017}$

**2. Carbonic Acid.**—10 c.c. of liquor are diluted to 400 c.c., 10 c.c. of ammoniacal calcium chloride (1 c.c. = .044 gram  $\text{CO}_2$ ) added, and the whole heated in a stoppered bottle for one and a half to two hours in a water bath at 100 degrees Cent. Cool somewhat, filter, wash by decantation with boiling water, and dissolve the calcium carbonate in 25 c.c. to 50 c.c.  $\frac{\text{N}}{2}$  HCl, with added cold water to prevent loss of acid. The small amount of calcium carbonate on the filter paper is best recovered by incineration.

$\text{CO}_2$  grams per 100 c.c. of liquor =  $.011 \times 10 \times \text{c.c.} \frac{N}{2}$  acid

$$\text{H.E.} = \frac{\text{CO}_2 \text{ grams}}{.022}.$$

3. **Chloride.**—10 c.c. of boiled liquor (for convenience 250 c.c. are boiled, to expel sulphide, &c., cooled and made up to 250 c.c. for estimation of chloride, sulphocyanide, ferrocyanide, &c.) are diluted to 150 c.c., 20 c.c. of hydrogen peroxide (10 volumes free from chloride) added, and the solution boiled until the brown colour has almost entirely disappeared; 10 to 15 drops of potassium chromate solution are then added to destroy the excess of peroxide and to aid in the removal of organic matter, and the boiling continued for five minutes. Filter, if necessary, from traces of green chromium hydrate, cool, neutralise by addition of a pinch of sodium bicarbonate, and titrate with  $\frac{N}{10}$   $\text{AgNO}_3$ .

*Calculation—*

$$\text{HCl grams per 100 c.c.} = .00364 \times 10 \times \text{c.c.} \frac{N}{10} \text{AgNO}_3.$$

4. **Sulphur**—(A) *As Sulphate.*—250 c.c. of the liquor are concentrated to about 10 c.c. on the water bath, 2 c.c. of strong hydrochloric acid added, and the evaporation continued to dryness to decompose thiosulphate and render organic matter less soluble in water. The residue is extracted with water and the filtered solution made up to 250 c.c. The sulphate is determined by precipitating 100 c.c. of this solution with barium chloride, allowing the precipitate one night to settle. The amount of oxidation undergone by the thiosulphate under these conditions is insignificant.

*Calculation—*

$$\text{Sulphur as sulphate, grams per 100 c.c.} = 0.1373 \times \text{grams BaSO}_4.$$

(B) *As Sulphocyanide*.—To 50 c.c. of the boiled solution (see "Chloride," as above) add ferric chloride, in amount slightly in excess of that required to complete the precipitation of the ferrocyanide (*Note*.—As ferrocyanide has only been detected in gas liquor analysed here on very rare occasions, addition of ferric chloride is generally found to be unnecessary, and is omitted. The appearance of Prussian blue, however, on adding ferric chloride (slightly acid) to the boiled liquor is regarded as one of the best qualitative tests for ferrocyanide) as Prussian blue, filter (the solution may be warmed to promote separation of the blue in the flocculent condition essential for rapid filtration), cool, add sulphuric acid in sufficient excess, followed by copper sulphate, and set aside in stoppered flask for one or two hours in the cold to deposit the cuprous salt. Filter cold, wash thoroughly with hot water, using a little sodium sulphate in the wash water if the precipitate shows a tendency to pass through the filter paper; the final washings must remain colourless on addition of a trace of ammonium sulphide. Wash the cuprous sulphocyanide, which should be white, back into the flask, the last traces being removed from the paper by warming on a clock glass with dilute nitric acid (1:3), add 1 c.c. to 2 c.c. of strong nitric acid, and boil the solution until green (in presence of much organic matter evaporation to dryness and gentle ignition, followed by further treatment with nitric acid, is sometimes required to complete the oxidation of the copper). Cool the oxidised liquid, add slight excess of sodium carbonate, acidify with acetic acid, add potassium iodide, dilute and titrate the liberated iodine with  $\frac{N}{10}$  thiosulphate, using starch as indicator.

#### Calculation.—

Sulphur as sulphocyanide, grams per 100 c.c.  $2 \times .00312 \times$   
 c.c.  $\frac{N}{10}$  thiosulphate.

(C) *As Sulphide, Sulphite, and Thiosulphate.*—(1.) 10 c.c. of liquor are diluted to 500 c.c., acidified with hydrochloric acid, and titrated with  $\frac{N}{10}$  iodine, starch as indicator.

The volume of  $\frac{N}{10}$  iodine required determines that of the liquor taken for (2.).

(2.) 10 c.c. of liquor, or more, are added to excess of ammoniacal zinc chloride solution, diluted to about 80 c.c. with warm water, filtered, and thoroughly washed with warm water (about 40 to 50 degrees Cent.).

(a) *Sulphide.*—The zinc sulphide on the filter is washed into excess of  $\frac{N}{10}$  iodine, acidified with hydrochloric acid (the last traces of sulphide being washed through with cold dilute acid) after vigorous agitation to complete the solution of zinc sulphide, water is added, and the excess iodine determined with  $\frac{N}{10}$  thiosulphate.

*Calculation—*

Sulphur as sulphide, grams per 100 c.c. =  $10 \times .0016 \times \text{c.c.}$   
 $\frac{N}{10}$  iodine.

$$\text{H}_2\text{S} = 10 \times .0017 \times \text{c.c.} \frac{N}{10} \text{ iodine.}$$

$$\text{H.E.} = \frac{\text{H}_2\text{S grams}}{.017}.$$

(b) *Sulphite and Thiosulphate.*—The conclusion is reached that no exact estimation of sulphite and thiosulphate is possible in ammoniacal liquor by any method based on titration with  $\frac{N}{10}$  iodine, except in quite exceptional cases.

A united figure for these two constituents can be reached by difference, subtracting from the total sulphur found by bromine oxidation the sum of the sulphurs present as sulphate, sulphocyanide, and sulphide.

(D) *Total Sulphur*.—50 c.c. (100 c.c. of weaker liquors) of liquor are delivered drop by drop from a burette into a flask containing excess of bromine (free from sulphur), covered by water, strongly acidified by hydrochloric acid; the oxidised solution is evaporated to dryness on the water bath, the residue repeatedly extracted with boiling water, filtered, cooled, made up to 250 c.c., and 100 c.c. precipitated with barium chloride.

*Calculation*—

Sulphur grams per 100 c.c. =  $5 \times .1373 \times \text{grams BaSO}_4$ .

In the case of some liquor, *e.g.*, coke oven liquor, oxidation with bromine is often found to yield a heavy yellow precipitate of brominated phenols; this may retain traces of sulphur in amount sufficient to affect the percentage distribution sulphur figures unless it is recovered by fusion with potassium carbonate and nitrate in the total. The correctness of the bromine oxidation method is confirmed later.

(E) *Sulphur as Polysulphide*.—The conclusion is reached that the methods of analysis elaborated so far afford no certain evidence of the existence of polysulphide in ammoniacal liquor; indeed, the known reactions of this body with sulphite to form thiosulphate, and with cyanide to form sulphocyanide, appear to exclude the possibility of its occurrence in ordinary ammoniacal liquor.

The more important papers relating to the above are:—

“Examination of the Ammoniacal Liquor of the Gasworks,” by S. Dyson (*Journal, Society of Chemical Industry*, 1883, pp. 229-231).

(*Note*.—Dyson procedure for estimation of sulphocyanide is regarded as untrustworthy.)

“Some Notes on Gas Liquor and Ammonia Purification” (in which methods of stating results are specially detailed), by Lewis T. Wright (*Journal, Society of Chemical Industry*, 1886, pp. 655-661).

“Laboratory Notes,” presented to the Incorporated Gas

Institute (35th Annual Meeting), by John T. Sheard (*Journal of Gas Lighting*, July 1898).

"Coal-Tar and Ammonia," by George Lunge (3rd edition, pp. 738-755).

"Volumetric Analysis," by Francis Sutton (8th edition, pp. 78-87).

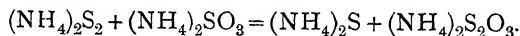
For "Estimation of Chloride" see Abstract of paper by O. Herling (*Journal, Society of Chemical Industry*, 1900, p. 336).

"Estimation of Cyanogen Compounds," by W. Feld (*Jour. für Gasbeleuchtung*, 1903, 46 (29) to (33), pp. 561-666).

### Estimation of Sulphite in Ammoniacal Liquors.—

The difficulties attending an exact estimation of sulphite in ammoniacal liquor have been fully considered, in the hope of arriving at a reliable method. Numerous methods were tried, and the following adopted:—

*Polysulphide Method.*—It is a well-established fact that solutions of ammonium polysulphide and ammonium sulphite react to form ammonium sulphide and ammonium thiosulphate, *e.g.*:—



As a result of this reaction the iodine value of the solution is decreased by 1 c.c.  $\frac{N}{10}$  iodine for every .0032 gram of sulphur as sulphite decomposed, the iodine value of the thiosulphate produced being half that of the sulphite reduced. This decrease affords a means, therefore, of calculating the amount of sulphite present.

The procedure adopted is as follows:—

(a) Total iodine value of solution is determined by titrating 10 c.c. of diluted and acidified liquor with  $\frac{N}{10}$  iodine.

$\frac{N}{10}$  iodine equivalent of  $\text{H}_2\text{S} + \text{H}_2\text{SO}_3 + \text{H}_2\text{S}_2\text{O}_3 = \text{A c.c.}$



(b) Iodine equivalent of sulphide is determined by ammoniacal zinc chloride method described above.

$$\frac{N}{10} \text{ iodine equivalent of } H_2S = B \text{ c.c.}$$

$$\frac{N}{10} \text{ iodine equivalent of } H_2SO_3 + H_2S_2O_3 = A - B.$$

(c) Iodine equivalent of half the sulphite and the thio-sulphate is determined by adding 10 c.c. of liquor to 10 c.c. or more of diluted polysulphide liquor (prepared by digesting strong ammonium sulphide solution with powdered sulphur, decanting; 2 c.c. of this solution freshly diluted to 100 c.c. gives the dilute solution used for analysis). After standing five to ten minutes in the cold, the clear, yellow liquor is precipitated by excess of ammoniacal zinc chloride and filtered; the filtrate is acidified with hydrochloric acid and titrated with  $\frac{N}{10}$  iodine.

$\frac{N}{10}$  iodine (less iodine for thiosulphate contained in the polysulphide used) is the equivalent of  $\frac{1}{2} H_2SO_3 + H_2S_2O_3$ .

Two examples will make this clear.

1. *Coke Oven Liquor* :—

10 c.c. liquor	$H_2S + H_2SO_3 + H_2S_2O_3$	8.57 c.c.	$\frac{N}{10}$ iodine	A.
"	$H_2S$	7.71	"	B.
"	$H_2SO_3 + H_2S_2O_3$	0.86	"	A - B.
"	$\frac{1}{2} H_2SO_3 + H_2S_2O_3$	0.60	(thiosulphate in polysulphide deducted) C.	
"	$\frac{1}{2} H_2SO_3$	0.26	"	A - (B + C).

Whence

Sulphur as sulphide =  $10 \times .0016 \times 7.71 = .1234$  gram of sulphur per 100 c.c.

Sulphur as sulphite =  $10 \times .0016 \times 2 \times .26 = .0083$  gram of sulphur per 100 c.c.

Sulphur as thiosulphate =  $10 \times .0064 \times .34 = .0218$  gram of sulphur per 100 c.c.

2. *Gas Liquor* (cyanide present, hence polysulphide presumably absent) :—

10 c.c. liquor	$\text{H}_2\text{S} + \text{H}_2\text{SO}_3 + \text{H}_2\text{S}_2\text{O}_3$	$= 32.13$	A.
„	$\text{H}_2\text{S}$	$= 30.71$	B.
„	$\text{H}_2\text{SO}_3 + \text{H}_2\text{S}_2\text{O}_3$	$= 1.42$	A - B.
„	$\frac{1}{2}\text{H}_2\text{SO}_3 + \text{H}_2\text{S}_2\text{O}_3$	$= 1.33$	C.
„	$\frac{1}{2}\text{H}_2\text{SO}_3$	$= .09$	A - (B + C).

Whence

$$\begin{aligned}\text{Sulphur as sulphide} &= 10 \times .0016 \times 30.71 = .4914 \text{ gram.} \\ \text{„ sulphite} &= 10 \times .0016 \times 2 \times .09 = .0029 \text{ gram.}\end{aligned}$$

Sulphite, if present, only exists in this liquor in an insignificant amount. It is reasonable to conclude that it is absent. It should be pointed out that the iodine figure for  $\frac{1}{2}\text{H}_2\text{SO}_3$  is reached by subtraction = A - (B + C). This will eliminate, to a material extent, the effect due to interference of organic matter, as titrations A and C are carried out under very similar conditions, while B is not considered to be materially affected under the conditions observed for the precipitation of the zinc sulphide.

Careful experiments were also made on mixtures containing various known proportions of sulphite and thio-sulphate, both in presence and absence of organic matter, to determine the limitations of this method.

The results obtained are shown in the accompanying Table, which, it is hoped, is sufficiently clearly set out to save detailed explanation.

In experiments Nos. 7 and 8 the results of analysis clearly indicate absence of sulphite in the solution examined. In experiments Nos. 1 to 6 the amount of sulphite found in each case is in satisfactory agreement with that taken.

# DISTRIBUTION OF SULPHUR IN AMMONIACAL LIQUORS. 129

## ESTIMATION OF SULPHITE BY POLYSULPHIDE METHOD. (PER 100 C.C.)

Liquor.	Sulphur, as Poly- sulphide taken for analysis.	Taken Sulphur, Grams.					Found Sulphur, Grams.		
		As Sulphide.	As Thio- sulphate.	As Sulphite.		As Sulphide.	As Thio- sulphate.	As Sulphite.	
				Present.	Added.				Total.
<i>A. Organic matter absent.</i>									
No. 1	.0420	absent	absent	.0156	...	.0156	...	...	.0154
No. 2	.0286	"	.0384	.0104	...	.0104	...	.0392	.0102
<i>B. Organic matter present.</i>									
Coke oven liquor									
June 23, No. 3	.0715	...	...	...	...	...	.1234	.0218	.0084
With added sul- phite, No. 4	.0715	.1234	.0218	.0084	.0362	.0445	.1202	.0282	.0304
June 24, No. 5	.0715	...	...	...	...	...	.1218	.0198	.0075
With added sul- phite, No. 6	.0715	.1218	.0198	.0075	.0259	.0334	.1206	.0260	.0301
Polysulphide added to re- move sulphite, No. 7	.0715	.1368	.0413	absent	...	absent	.1362	.0378	.0010
Gas liquor (oxidized by partial expo- sure to air, 20 months), No. 8	.2145	absent	...	...	...	absent	.2204	...	.0004

\* Exclusive of that reacting as sulphide.

**Distribution of Sulphur in Ammoniacal Liquors.**—If we subtract from the total sulphur found by bromine oxidation the sum of the constituent sulphurs found as sulphate, sulphocyanide, "thiosulphate" (determined by titration of the acidified filtrate from the sulphide by N<sub>2</sub> iodine), and sulphide, a difference in figure is obtained, which has proved to be invariably negative in sign, since the adoption of the improved method of estimating sulphocyanide described above.

Evidence was previously obtained that some of the difference noticed was due to the presence of sulphite, which caused the "thiosulphate" figure to largely exceed its proper value by reason of the factor for conversion of  $\frac{N}{10}$  iodine into sulphur as thiosulphate being .0064 gram

sulphur per 1 c.c.  $\frac{N}{10}$  iodine, while that for sulphite is only one-fourth of this. This has failed, however, to reduce the difference figure to the limits of reasonable experimental error, and the conclusion is reached, therefore, that organic matter is the disturbing cause, as such differences are not noticed when the same methods are applied to determine the same constituent in solutions from which organic matter is excluded.

Various considerations point to the thiosulphate titration as the one peculiarly liable to such interference; for this reason the iodine method of estimating this constituent in ammoniacal liquors is finally rejected in favour of a figure arithmetically obtained by difference.

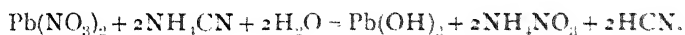
**Estimation of Cyanogen Compounds in Ammoniacal Liquor.**—In ammoniacal liquor three cyanogen compounds alone are of technical interest: cyanide (hydrocyanic acid), ferrocyanide, and thiocyanate (sulphocyanide).

In these analyses the "total cyanogen" contents of any given liquor is taken to be the sum of the hydrocyanic acid (HCy) equivalents, grams per 100 c.c. of the cyanide, ferrocyanide, and thiocyanate present; and the "cyanogen distribution" figure for each of the compounds named to be the hydrocyanic acid equivalent of the compound per 100 parts of "total cyanogen" (as HCy) present. The methods are:—

*Cyanide (Hydrocyanic Acid).*—Feld's method for the estimation of ammonium cyanide is based upon the fact that if alkaline cyanides are distilled with a solution of magnesium chloride, or lead nitrate, the cyanogen is expelled

quantitatively as hydrocyanic acid. If sulphide be present in the solution to be distilled, lead nitrate should be used to avoid the evolution of sulphuretted hydrogen which occurs when magnesium chloride is employed.

The reaction when lead nitrate is used may be represented by the equation :—



In applying this method for the estimation of hydrocyanic acid, it has been found convenient to employ the apparatus used for determination of ammonia, both free and fixed. This consists of a 500 c.c. round-bottomed flask provided with two-holed caoutchouc stopper with inlet tube sealed in liquor, and exit tube connected through a catch bulb to Liebig's condenser and receiver.

It is safer, in distilling off hydrocyanic acid, to seal exit tube of the condenser in the 25 c.c. of  $\frac{\text{N}}{\text{I}}$  caustic soda, with which the receiver is charged, a precaution quite unnecessary when ammonia is being estimated.

#### *Example—*

50 c.c. of liquor are distilled with excess of a 20 per cent. solution of lead nitrate (50 to 100 c.c.) into 25 c.c. of  $\frac{\text{N}}{\text{I}}$  soda for twenty-five minutes, the distillate diluted to about 400 c.c., a crystal of potassium iodide (about 0.2 gram) added, and the solution titrated on  $\frac{\text{N}}{10}$  silver nitrate :—

$$(1 \text{ c.c.} = .0054 \text{ gram HCN})$$

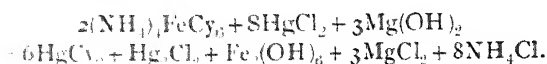
$$\frac{\text{N}}{10} \text{ AgNO}_3 = 6.30 \text{ c.c.}$$

Whence

$$\text{HCN per 100 c.c. liquor} = 2 \times 6.30 \times .0054 = .068 \text{ gram.}$$

$$\text{Hydrogen equivalent (H.E.)} = \frac{.068}{.027} = 2.5.$$

*Estimation.*—Feld estimates this compound by boiling in alkaline solution with mercuric chloride, whereby mercuric cyanide is formed; the latter body is then decomposed with sulphuric or hydrochloric acid, the hydrocyanic acid liberated, being distilled off, and titrated with silver nitrate as mentioned above. Magnesium chloride is added to the alkaline solution before addition of mercuric chloride, to prevent precipitation of mercuric oxide. The reaction in the case of ammonium ferrocyanide may be represented as follows:—



The distillation of the mercuric cyanide is conveniently carried out in the same apparatus as that employed for the estimation of hydrocyanic acid (as above), procedure, as regards the collection and titration of the evolved hydrocyanic acid, being in both cases identical. The use of the same apparatus for estimation of "free" and "fixed" ammonia, hydrocyanic acid, and ferrocyanide is an advantage much appreciated in the complete examination of ammoniacal liquors, where analyses have to be promptly made to minimise the changes in composition that rapidly follow on exposure to the air.

The volume of liquor taken is 250 c.c. Solution is boiled to expel volatile salts, a moderate excess (10 to 15 c.c.) of 6 N. caustic soda added (amount calculated from the "fixed" ammonia found), and the boiling continued for a further period of fifteen minutes to remove fixed ammonia, and bring separated ammonium ferrous ferrocyanide into solution. Cool, make up to 250 c.c.

To 50 c.c. of the solution diluted to 100 to 150 c.c., and raised to boiling, add 5 c.c. of 6 N. soda (NaOH) followed by 30 c.c. of 3 N. magnesium chloride (add slowly to avoid formation of clots), boil for five minutes, add 25 to 50 c.c.

a boiling solution of  $\frac{\text{N}}{10}$  mercuric chloride, and continue

the boiling for not more than ten minutes. The liquor is then distilled for twenty minutes with 30 c.c. of 6 N. sulphuric acid into 25 c.c. of  $\frac{N}{2}$  caustic soda. To the distillate add a pinch of lead carbonate, and filter off the precipitated lead sulphide, dilute to about 400 c.c., add a crystal of potassium iodide and titrate on  $\frac{N}{10}$   $\text{AgNO}_3$  (1 c.c. = .0054 gram HCy).

The presence of ferrocyanide in ammoniacal liquor is so uncertain that it is desirable to identify it by qualitative test before embarking on the somewhat lengthy procedure described above. For this purpose it is sufficient to add to the acidified boiled liquor a drop of dilute ferric chloride and gently warm.

(The boiled liquor sometimes contains small traces of separated ammonium ferrous ferrocyanide; it must therefore be well shaken before applying the Prussian blue test for ferrocyanide. Addition of peroxide of hydrogen to the boiled liquor in such cases produces a blue body by oxidation.)

If ferrocyanide is present the red colour of the thiocyanate changes to blue, and in cases where sufficient is present, a flocculent blue precipitate separates.

*Thiocyanate (Sulphocyanide)*—(A) *Ferrocyanide absent.*—If qualitative test (ferric chloride to acidified boiled liquor and warming) indicates that ferrocyanide is absent, add to 50 to 100 c.c. of the boiled liquor acid sulphite of soda containing free sulphurous acid in excess, followed by copper sulphate, and set aside in closed flask for one and a half to two hours in a warm place (25 to 30 degrees Cent.) to deposit the cuprous salt. This procedure ensures separation of the cuprous thiocyanate in a form readily retained on filtration through any good filter paper suitable for use with barium sulphate precipitate. Filter, wash thoroughly with hot water; the final washing must remain colourless on addition of a drop of dilute potassium ferrocyanide, or

ammonium sulphide. Wash the cuprous thiocyanate which should be white (grey if organic matter is present) into a platinum dish, the last traces being removed by warming the filter paper on a clock glass with dilute nitric acid (1 : 3), add 1 c.c. of strong nitric acid, and evaporate to dryness on the water bath. The residue is gently ignited to remove organic matter, moistened with nitric acid, and again cautiously ignited to expel oxides of nitrogen; dissolve in dilute sulphuric acid (normal strength is suitable), add a slight excess of carbonate or bicarbonate of soda added and the separated carbonate dissolved in acetic acid. To the clear green solution of the acetate add excess of potassium iodide, and titrate the liberated iodine with  $\frac{N}{10}$  thiosulphate, using starch indicator (added towards the end of the titration). In general, the reaction proceeds a little sluggishly at the finish. Addition of thiosulphate should be added until the blue colour ceases to return after the solution has stood for a minute. It is more satisfactory to add a slight excess of  $\frac{N}{10}$  thiosulphate and bring back with  $\frac{N}{10}$  iodine. For technical purposes, evaporation of the cuprous salt to dryness with nitric acid may be omitted as an unnecessary refinement. In this case the cuprous salt should be washed back into the flask and oxidised there by boiling for fifteen to twenty minutes with 2 c.c. of concentrated nitric acid. The free acid is then neutralised by sodium carbonate, and the solution prepared for titration on  $\frac{N}{10}$  thiosulphate in the manner described above.

Results so obtained are approximately accurate, but the complete oxidation of the cuprous salt appears to be somewhat difficult to complete in the case of some liquor and, in addition, the end point is less easy to determine with certainty when nitrates and organic compounds are both present. Thus :—



Organic matter absent—

Thiocyanate taken	-	-	-	-	-	10.00	$\frac{N}{10}$	c.c.
Found (1) Nitric acid solution evaporated to dryness, &c.	-	-	-	-	-	9.98		c.c.
(2) Nitric acid solution boiled 15 to 20 minutes	-	-	-	-	-	9.98		„

Organic matter present—

Coke oven liquor—Thiocyanate found, grams sulphur per 100 c.c.

(1) Nitric acid solution evaporated to dryness, &c.	-	-	-	-	{ (a) .0260 } { (b) .0269 }	.0264
(2) Nitric acid solution boiled 15 to 20 minutes	-	-	-	-	-	.0278

Oxidation of the dried cuprous thiocyanate by direct ignition, followed by treatment with nitric acid in platinum dish, is inadmissible; considerable traces of copper appear to be volatilised on ignition of the precipitate in contact with filter paper. Thus:—

Organic matter absent—

Thiocyanate taken	-	-	-	-	-	Grams Sulphur.	
„ found by ignition method	-	-	-	-	{ (a) .0139 } { (b) .0142 }	.0160 .0140	Loss = 12 per cent.

Organic matter present—

Thiocyanate found, grams sulphur per 100 c.c.

(1) Nitric acid solution evaporated to dryness, &c.	-	-	-	-	.0683	} Loss = 7 per cent.
(2) Precipitate ignited to oxide, then evaporated with $\text{HNO}_3$ &c.	-	-	-	-	.0632	

(B) *Ferrocyanide present.*—To remove the ferrocyanide, add to the slightly acidified boiled liquor sufficient excess of ferric chloride solution to render it decidedly red (ferric thiocyanate) and warm gently (40 to 50 degrees Cent.) to render the “Prussian blue” flocculent; filter. To the filtrate, cooled, add acid sulphite of soda containing free sulphurous acid and proceed as described in (A).

This procedure ensures the separation of the whole of the ferrocyanide. If desired, the "Prussian blue" thus obtained can be used for the approximate estimation of ferrocyanide in the gas liquor by treatment with caustic soda and distillation by Feld's method. Thus:—

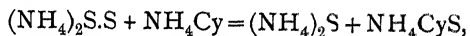
Ferrocyanide, calculated as HCy, grams per 100 c.c.

	Gasworks Liquor.	Coke Oven Liquor.
(1) Feld's method applied direct to boiled liquor	0.172	.0065
(2) Feld's method applied to "Prussian blue" separated by ferric chloride	0.161	.0065

The conversion of sulphide into thiosulphate is already well known, but the conversion of hydrocyanic acid into thiocyanate in presence of ammonium sulphide is of equal importance, but less widely known. A single example will serve to illustrate the point. Thus, a sample of coke oven liquor was examined as received, and again after exposure to an equal volume of air in stoppered bottle for fifty-eight days, with the following results:—

	When Opened.	After 58 days' Exposure to Air.
Cyanide, grams HCy per 100 c.c.	.053	.024 - .029
Thiocyanate, grams HCy per 100 c.c.	.002	.026 + .024
Ferrocyanide, grams HCy per 100 c.c.	nil	nil
	<hr/> 0.055	<hr/> .050

Allowing for slight loss of hydrocyanic acid by volatilisation into the air above the liquor, we see that 50 per cent. of the cyanide has been converted into thiocyanate by simple contact with air in the cold; a corresponding conversion of "free" ammonia  $((\text{NH}_4)_2\text{S})$  into "fixed" ammonia  $(\text{NH}_4\text{CyS})$  has accompanied the change. The reaction doubtless proceeds on the lines:—



the polysulphide being itself obtained by direct oxidation of ammonium sulphide.

Cyanide and polysulphide, therefore, react to form thiocyanate, and excess of either body implies absence of the other. This fact accounts for the absence of polysulphide from gasworks and coke oven liquors, where excess of cyanide is generally present.

## CHAPTER VIII.

### *ANALYSIS OF OXIDE OF IRON.*

IN the analytical valuation of a natural oxide of iron, or bog-ore, it is necessary to estimate the moisture, the organic matter or peat, the ferric oxide, and silicious matter. These analyses are all comparatively simple, with the exception of the ferric oxide; this, as will be explained later, is the most important compound in the material, and it is not only necessary to know the amount of ferric oxide, but to know the exact state this exists in the bog-ore.

What is wanted in a good bog-ore for gas purification is that the ferric oxide should be in a hydrated state. There is no doubt that there is more than one compound of hydration of oxide of iron in a bog-ore, and it would be as well to give here the known compounds, viz. :—

Gothite,  $\text{Fe}_2\text{O}_3, \text{H}_2\text{O}$ .  
Limonite,  $2\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ .  
Xonthosiderade,  $\text{Fe}_2\text{O}_3, 2\text{H}_2\text{O}$ .  
Bog-ore,  $\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ .

Exactly how many or how few of these hydrated compounds of iron exist in a bog-ore has never been definitely settled. It is the usual method to calculate it as all existing in the one form, viz.,  $\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ . It is really immaterial which is taken, so long as a definite arrangement is arrived at, so that there can be no disputes.

There are three known oxides of iron, and these are :—

1. Ferrous oxide,  $\text{FeO}$ .
2. Ferric oxide,  $\text{Fe}_2\text{O}_3$ .
3. Ferroso-ferric oxide, or magnetic oxide,  $\text{Fe}_3\text{O}_4 = \text{FeO}, \text{Fe}_2\text{O}_3$ .

Of these, the only one that is supposed to exist in a natural bog-ore is the ferric oxide, although at times one comes across a sample that certainly contains some other oxide of iron, which is not the ferric oxide.

In a chemical analysis on an oxide it is most difficult to ascertain in which form the oxide exists, and it is general to assume it to exist as  $\text{Fe}_2\text{O}_3$ , but it is also necessary to know whether this oxide is in the proper state of hydration, and the only method to adopt is to submit the sample to a practical test, viz., foul it. This will certainly show the quality of the oxide, and whether the material is suitable for purification. We will now proceed to the analysis of a sample of oxide.

**Moisture.**—The sample is well mixed and ground in a mortar; 10 grams are weighed out into a counterpoise platinum crucible or basin. This is then put into a water oven which is kept at 212 degrees Fahr. (100 degrees Cent.) for twenty-four hours. The crucible and contents are weighed again, and then replaced in the oven again for a further period of six hours, and then reweighed. If there is no decrease in weight the amount of loss represents the water in sample; if there is a further loss the operation must be repeated until no further loss takes place.

*Example—*

Crucible	46.907 grams.
Oxide	10.000 "
Total	56.907 "
Weight after drying for 24 hours	52.185 "
	4.722 grams.
Weight after drying for a further period of 6 hours	52.183 "
	4.724 grams.

After this the weight was constant.

Therefore the sample contains  $4.724 \times 10 = 47.24$  per cent. of water at 212 degrees Fahr.

**Organic Matter.**—The crucible and contents from the above are now ignited in a muffle furnace, and it is desirable to always have the same method, so as to have uniformity in the results. The loss in weight is organic matter and combined water.

*Example—*

Weight of crucible from—

Last experiment	-	-	-	-	52.183	grams.
After ignition	-	-	-	-	50.423	„
					<hr/>	
					1.760	grams.

Organic matter (peat) and combined water = 17.60 per cent.

**Ferric Oxide.**—The residue, after organic matter has been burnt off, is now dissolved in boiling HCl acid (50 per cent. solution) until all the ferric oxide is dissolved, judged by the residue being free from colour.

The solution is then diluted and filtered, the insoluble residue and filter paper being afterwards ignited and weighed, which gives us the percentage of silicious matter.

The filtrate is then treated as follows for the ferric oxide (there are numerous methods for the estimation of the ferric oxide, but the two principal ones will only be described here):—

(1.) A portion of the filtrate is taken and precipitated while hot with NaOH, to eliminate the alumina present; the precipitated ferric hydrate is filtered and washed with hot water; the ferric hydrate is redissolved in hydrochloric acid, and reprecipitated with  $\text{NH}_3$ . This is filtered, washed with hot water, ignited, and weighed.

The ignition must be made with the cover on the platinum crucible, as during the transition of the ferric hydrate to anhydrous ferric oxide a certain loss will be occasioned by “decrepitation,” *i.e.*, a certain portion of the precipitate will be projected from crucible and lost. The crucible and contents are now placed in a desiccator to cool, and weighed.

The increase in weight from first weight of crucible gives the amount of ferric oxide in the amount of liquor taken.

*Example—*

Filtrate and washing made up to 500 c.c.

∴ 500 c.c. = 10 grams of material.

50 c.c. = 1 gram of material.

25 c.c. = 0.5 " "

25 c.c. are taken and precipitated with NaOH, &c.

Crucible - - - - 46.907

Crucible + residue - - - 47.071

---

.164 gram  $\text{Fe}_2\text{O}_3$  in 0.5 gram.

200

---

32.800  $\text{Fe}_2\text{O}_3$ .

Now 160 parts of  $\text{Fe}_2\text{O}_3$  give 214 part of  $\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ ; therefore having obtained the percentage of  $\text{Fe}_2\text{O}_3$ , multiply by 1.3375 will give the percentage of  $\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ .

$32.80 \times 1.3375 = 43.86$  per cent.  $\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ .

(2.) By titrating with a standard solution of potassium dichromate. This solution is made by exactly weighing 4.913 grams of pure potassium dichromate (previously dried by being gently fused in a porcelain crucible and then finely powdered in a dry mortar), dissolved in water in a litre flask, and then the solution made up to 1000 c.c. with distilled water at 60 degrees Fahr. (or 15 degrees Cent.).

One litre of this  $\frac{\text{N}}{10}$  solution contains one-tenth of an equivalent of available oxygen in grams, *i.e.*, 0.8 gram; therefore 1 c.c. = 0.008 gram of available oxygen, and is therefore equivalent to 0.0056 Fe. Therefore the  $\frac{\text{N}}{10}$  potassium dichromate solution is as follows:—

1 c.c. = 0.0056 Fe.

1 c.c. = 0.0072  $\text{FeO}$ .

1 c.c. = 0.008  $\text{Fe}_2\text{O}_3$ .

1 c.c. = 0.0089  $\text{Fe}_2\text{O}_3, \text{H}_2\text{O}$ .

1 c.c. = 0.0107  $\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ .

The potassium dichromate solution is stable, and has no action on rubber. This solution must be "standardised," that is, its exact strength must be definitely ascertained; this is carried out by dissolving pure iron wire in dilute sulphuric acid, with the exclusion of air, in the apparatus shown in Fig. 35.

Take 0.5 gram of the fine iron wire used for binding flowers (which contains 99.6 per cent. Fe). About 100 c.c. of dilute sulphuric acid (1 in 5) are placed in the flask (which should have a capacity of about 250 c.c.), fitted

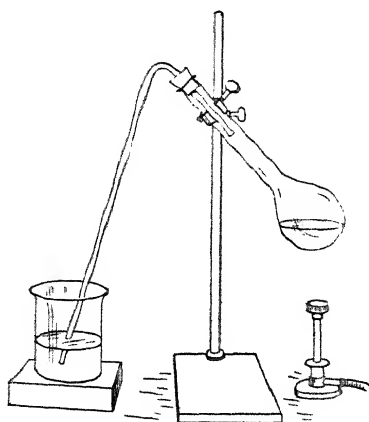


FIG. 35.—APPARATUS FOR DISSOLVING IRON ORES.

with a rubber cork and bent glass tube as shown in figure. The air in the flask is expelled by removing cork and dropping in two or three crystals of pure sodium carbonate; this expels all air, and as soon as the carbonate has dissolved, drop in the weighed quantity of iron. The cork is instantly inserted, and is as shown in figure, the bent tube dipping into a solution of sodium carbonate contained in the beaker. The

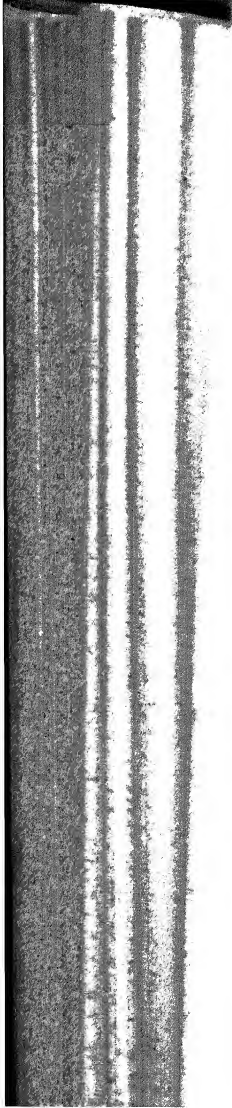
flask is gently heated by a small flame until the whole of the iron is dissolved. The gas is then withdrawn, and the flask allowed to cool. As the flask cools, the sodium carbonate solution contained in the beaker is drawn up into the flask. Directly the first drop enters the flask an effervescence of carbon dioxide is caused, which drives the liquid down again, at the same time filling the flask



when it has partially cooled in this water the cork is and air-free distilled water added; the flask is







1cc

3cc

10cc

15cc

20cc

25cc

30cc

35cc

COLOUR SLIPS SHOWING THE REDUCTION BY ZINC SULPHATE OF POTASSIUM DIOCHROMATE

then closed by a rubber stopper, and is held in a stream of cold water until quite cold; transfer to a 250 c.c. flask, measure it, and make up to the exact quantity with further addition of cold air-free water. 50 c.c. of this solution are then taken by means of a pipette and titrated with the dichromate solution, gradually added from a burette. The end of the reaction is ascertained by means of a freshly made solution of potassium ferricyanide used as indicator.

Drop reaction paper is cut into strips about 1 in. by  $\frac{1}{4}$  in., on this is put a little of the ferricyanide solution by means of a glass rod, and some of the iron solution put on the paper in the same way, so that when it spreads, it meets the ferricyanide and causes a blue line; but as the amount of ferrous salt is diminished by the gradual addition of the dichromate, the blue becomes less and less until finally the last drop so tested fails to give a blue colour. The tints shown in the Plate represent the gradual falling off in blue colour.

*Example—*

0.5 gram of iron dissolved in 250 c.c.  
0.1 „ „ in 50 c.c.

As the wire only contains 99.6 of iron, to find the exact weight multiply by 996.

Therefore 50 c.c. contain 0.0996 gram of iron. In titration it was found that 17.6 c.c. were used in the 50 c.c.

Therefore  $17.6 \times 5 = 88.0 \times .996 = 87.56$  c.c., and instead of the solution containing 0.5 gram in 250 c.c., it only contains 0.488 gram.

Therefore  $0.488 \div 87.56 = .005584$  instead of .0056 Fe.

Therefore the solution is slightly weaker than it should be, this is easily overcome by the use of a factor.

Having ascertained the exact strength of the decinormal solution it is now ready for use on the oxide of iron.

Proceed exactly in the same way as with the iron sulphate solution, but there is no need to wait for the same sample as was used for moisture, &c., but a fresh quantity

may be weighed, say 2 grams, dissolved in hydrochloric acid, in the flask as before. When all the iron has dissolved, filter, add a few pieces of pure granulated zinc, and boil until all the  $\text{Fe}_2\text{O}_3$  is reduced to  $\text{FeO}$ , which is shown by the solution going perfectly white, filter off the zinc, and make up to a definite volume.

A portion of this is then titrated with the potassium dichromate solution.

*Example—*

2 grams dissolved in hydrochloric acid, reduced by zinc, and made up to 400 c.c.

$$200 \text{ c.c.} = 1 \text{ gram.}$$

200 c.c. titrated with potassium dichromate; they required 41.0 c.c.

$$1 \text{ c.c. } \frac{N}{10} \text{ dichromate} = .008 \text{ Fe}_2\text{O}_3;$$

$$\therefore .008 \times 41 \times 2 = .656 \text{ gram Fe}_2\text{O}_3;$$

$$.656 \times 50 = 32.80 \text{ per cent. Fe}_2\text{O}_3;$$

or,

$$1 \text{ c.c. } \frac{N}{10} \text{ dichromate} = .0107 \text{ Fe}_2\text{O}_3, 3\text{H}_2\text{O};$$

$$\therefore .0107 \times 41 \times 2 = .8774 \text{ gram Fe}_2\text{O}_3, 3\text{H}_2\text{O},$$

$$.8774 \times 50 = 43.87 \text{ per cent. Fe}_2\text{O}_3, 3\text{H}_2\text{O}.$$

The insoluble residue, after dissolving the iron out, is now burnt off in a platinum crucible and weighed. Increase in weight =  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and inert matter.

*Example—*

Weight of crucible + insoluble residue	47.143
Crucible	46.907
	<hr/>
	.236

$$.236 \times 10 = 2.36 \text{ per cent. silica, alumina, \&c.}$$

We have now a complete analysis of a sample of oxide of iron, which is as follows:—

*Summary—*

Moisture	-	-	-	-	-	47.24	per cent.
Fe <sub>2</sub> O <sub>3</sub>	-	-	-	-	-	32.80	"
Organic matter, peat, and combined	-	-	-	-	-		
water	-	-	-	-	-	17.60	"
Silica, alumina, and inert matter	-	-	-	-	-	2.36	"
						<hr/>	
						100.00	

The Fe<sub>2</sub>O<sub>3</sub> = 32.80 per cent. expressed as being in the form of Fe<sub>2</sub>O<sub>3</sub>, 3H<sub>2</sub>O = 43.86 per cent.

It is usual to give the Fe<sub>2</sub>O<sub>3</sub>, 3H<sub>2</sub>O on the dry basis so that all analyses of various samples containing varying proportions of water are comparable.

The method of working from wet to dry basis is as follows:—

*Example—*

100.00 deduct 47.24 per cent. for water.  
47.24

52.76 equals dry material in 100 parts.

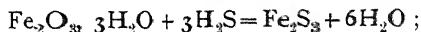
Therefore 52.76 contains 32.80 parts of Fe<sub>2</sub>O<sub>3</sub> or 43.86 parts of Fe<sub>2</sub>O<sub>3</sub>, 3H<sub>2</sub>O.

52.76 : 32.80 :: 100 : 62.16.

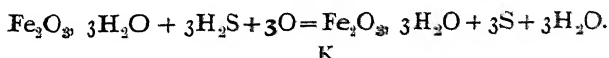
Therefore Fe<sub>2</sub>O<sub>3</sub> on dry basis is 62.16 per cent.

Therefore Fe<sub>2</sub>O<sub>3</sub>, 3H<sub>2</sub>O on dry basis is 82.92 per cent.

Having arrived at the analysis of this sample of oxide, it is now necessary to ascertain that the ferric oxide Fe<sub>2</sub>O<sub>3</sub> is in the proper state of hydration for the necessary chemical reaction that takes place in the purifiers, where the hydrate ferric oxide absorbs the sulphuretted hydrogen in the gas according to the following formula:—



or when revivification takes place *in situ*:—



Now in the analytical valuation of a bog-ore it is practically impossible to give any idea as to its value as a purifying reagent, *i.e.*, the ferric oxide may exist as a hydrate, or it may not, or in the analysis other oxides of iron may be estimated and expressed as existing in a state of hydration, which they do not. To form an exact idea what state the ferric oxide is in, the sample is subjected to an experimental fouling.

The method is as follows:—3 or 4 lbs. of the sample are taken and well mixed. A long glass cylinder (see Fig. 36) is now filled with the oxide, and crude gas is passed through it for twenty-four hours. The cylinder is now disconnected and put out in a porcelain tray or on a piece of wood and allowed to revivify, which usually takes from twenty to thirty hours. This is again made moisture and treated exactly the same as if on the large scale, and put into the cylinder and again subjected to the action of crude gas on the inlet to purifiers (the same place as before) for twenty-four hours when it is again revivified.

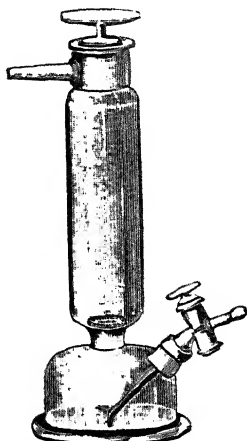


FIG. 36.—CYLINDERS FOR OXIDE OR WELDON MUD FOULINGS.

This operation is repeated till the sample fails to show any appreciable absorbing of sulphur, usually in about eight foulings.

A sample is taken from each fouling, the moisture is estimated, and the sulphur absorbed is extracted as in the valuation of spent oxide. I have found most excellent results by this method, especially if one has a standard to go by.

Take a sample of oxide that has been used on the works and one of which the fouling or absorbing abilities of sulphuretted hydrogen is known, and try it in this

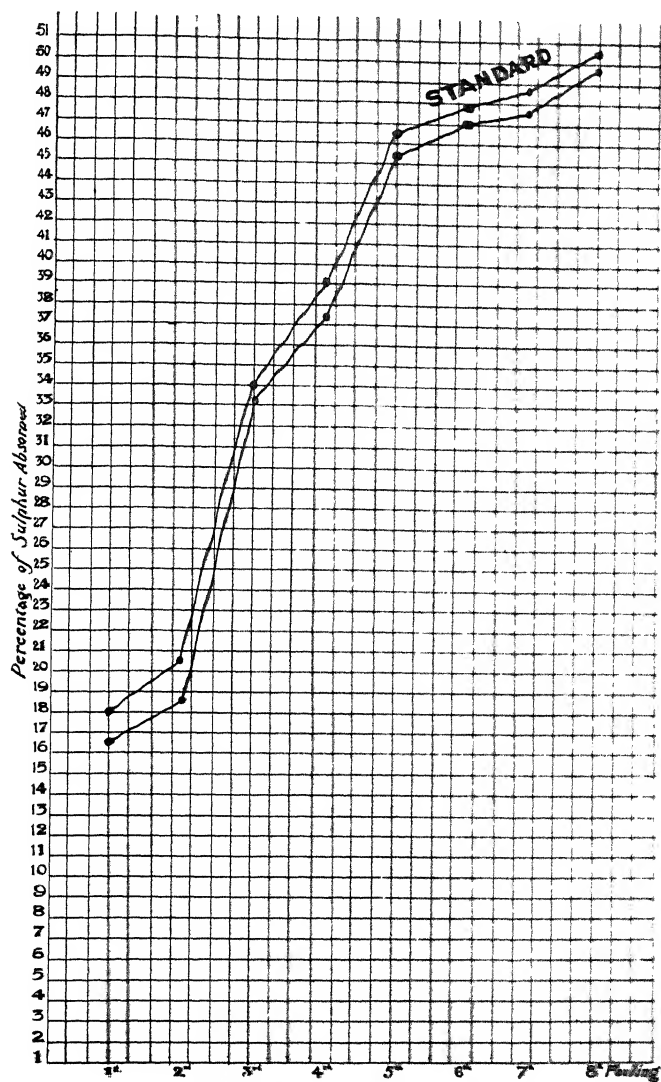


FIG. 37.—CHART SHOWING SULPHUR (DRY BASIS) ABSORBED BY OXIDE.

way. You will then have a standard by which you can compare any sample sent in, and if care is taken in the whole operation the result will be a most excellent criterion as to the suitability of an oxide for gas purification. The amount of sulphur absorbed is expressed in percentages on the dry basis.

The accompanying curves show the standard and sample, which are practically similar. In this method, one is able to form an opinion on any sample of oxide, and to know for certain what a given oxide is capable of doing when under actual practical working conditions.

In cases where the gas company require a high standard of ferric hydrate ( $\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ ), it is sometimes the practice to add various iron ores to the sample and bulk which are not hydrates, and which would require some considerable period before they would assume that state of hydration which would make them profitable to use in gas manufacture for the purification of gas from sulphuretted hydrogen. This addition is made because it is invariably the practice to estimate the total iron as ferric oxide, and to calculate all this as being in the form of the hydrate ( $\text{Fe}_2\text{O}_3, 3\text{H}_2\text{O}$ ).

To estimate the amount of ferrous oxide ( $\text{FeO}$ ) proceed exactly as in the method described for the standardising of the dichromate solution, taking very great care that no air is allowed to come into contact with the sample, else some of the  $\text{FeO}$  will be oxidised to  $\text{Fe}_2\text{O}_3$ , which will be estimated as  $\text{FeO}$ . Directly the sample is cool titrate with the bichromate solution, and if there is any, say for example 4 c.c. are used, this number of c.c. must be deducted from the total number of c.c. used in the reduction of the total iron as  $\text{Fe}_2\text{O}_3$ ,  $4 \times 2 = 8$  c.c., therefore this 8 c.c. must be deducted from the 82 c.c. mentioned before.

To estimate the total silica the method mentioned in the analysis of lime is used.

Having now described the usual method used in the



analysis of an oxide, it would be advisable here to draw attention to numerous experiments carried out by myself, some of which have been confirmed by Mr Leicester Greville, and described in the *Journal of Gas Lighting*, 14th September 1905.

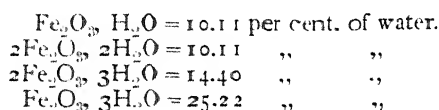
It was desired to ascertain for certain which was the correct formula for representing the hydrated ferric oxide either by the formula  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$ , or by the formula  $\text{Fe}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$ . 10 grams of the natural oxide which had previously shown good results by fouling were weighed out into a platinum dish. This was then put into a desiccator and constantly weighed until the weight was constant, showing that all the free moisture was absorbed by the sulphuric acid in the desiccator. The loss in weight was 40.37 per cent. The sample was then dried in the water oven at 100 degrees Cent. (212 degrees Fahr.) until the weight was constant; this gave a loss of 11.84 per cent.

Now the formula  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$  theoretically requires 10.11 per cent. of moisture. This practically proves that the monohydrate of iron,  $\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$  exists in the sample.

The sample was next placed in a combustion tube connected on to a calcium chloride tube which had been previously weighed, and the combined water which did not come off at 212 degrees Fahr. driven off together with the organic matter. This was weighed and showed 7.18 per cent. of combined water, and 23.42 per cent. organic matter.

To arrive at the exact amount of combined water the calcium chloride tube was connected up as before, and dry coal-gas passed through it which carried the combined water into another weighed calcium chloride tube. Now the combined water being 7.18 per cent., and the nearest hydrated oxide that we have to this is  $2\text{Fe}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ , which requires 10.11 per cent. of moisture.

Now the various hydrated oxides with the percentage of water theoretically required are :—



The method used was as follows :—The hydrate of iron having the formula  $\text{Fe}_2\text{O}_3, \text{H}_2\text{O}$  requires 10.43 per cent.  $\text{Fe}_2\text{O}_3$  to combine with it, and therefore as our water loss over sulphuric acid during desiccation may be taken as representing this hydrate, we shall require 10.43 per cent. of our total  $\text{Fe}_2\text{O}_3$  to combine with the water found, and  $10.43 - 32.80$  (our total Fe as  $\text{Fe}_2\text{O}_3$ ) will leave us 22.37 per cent.  $\text{Fe}_2\text{O}_3$ .

Now the combined water above 212 degrees Fahr. was found to be 7.18 per cent. We are justified in taking this as existing in the hydrate of  $2\text{Fe}_2\text{O}_3, 2\text{H}_2\text{O}$  (although some may exist as the other two hydrated oxides), and the  $\text{Fe}_2\text{O}_3$  required to give the formula  $2\text{Fe}_2\text{O}_3, 2\text{H}_2\text{O}$  requires 7.10 per cent.  $\text{Fe}_2\text{O}_3$ ; we have  $7.10 - 22.37$  per cent., leaving 15.27 per cent. of  $\text{Fe}_2\text{O}_3$  which we may call "unattached," or which exists in the sample as free  $\text{Fe}_2\text{O}_3$  unhydrated.

Other samples of well-known bog-ore confirm this result, although in some cases the "unattached"  $\text{Fe}_2\text{O}_3$  is very low, being as low as 1 to 2 per cent.

These experiments prove that there is more than one of the hydrates of iron present in a bog-ore. Undoubtedly the organic matter plays an important part; this organic matter is of an acid nature, and it is found that when ammonia (only a trace) is allowed to go forward into the purifiers they do better work, owing to the fact that the acid bases in the oxide are neutralised.

## CHAPTER IX.

### *NAPHTHALENE.*

BEFORE giving the various methods, &c., which have been tried to remove this compound, the methods for testing for it will first be considered. There are only two methods which have met with any amount of success, the first being that discovered by Dr Colman and J. F. Smith, and which is known as Colman and Smith Naphthalene Test, the second being that known as Dickenson-Gair's Test.

1. **Colman and Smith Naphthalene Test.** — This method was explained in a paper read by Dr Harold Colman before the Society of Chemical Industry in January 1900.

A solution of picric acid which is nearly saturated at normal temperature, and which is about  $\frac{1}{10}$  normal. The strength of this solution is accurately determined by titration with  $\frac{N}{10}$  soda solution, using lacmoid as indicator, the colour of which is changed from brownish yellow to green on a slight excess of alkali.

The apparatus consists of a series of five bottles, the first having a capacity of 4 oz., the second of 10 oz., the other three being of 2 oz. capacity each. They are charged as follows:—The first with a solution of citric acid which serves to remove any ammonia in the gas, the second contains 100 c.c. of the picric acid solution, the third and fourth 25 c.c. each of the same solution, whilst the fifth bottle serves to retain any splashing that may be carried

forward. The fifth bottle is connected to the meter. The various bottles are connected preferably with metallic flexible tubing, as ordinary rubber tubing absorbs naphthalene when new; if rubber tubing is used, the glass ends of the bottles must be brought as close together as possible, so that the gas comes in contact with the rubber as little as possible. The gas is now passed through the bottles at the rate of from 0.5 to 1.0 cub. ft. per hour until 10 cub. ft. have passed; the contents of the smaller bottles are washed into the 10-oz. bottle, using as little water as possible, as

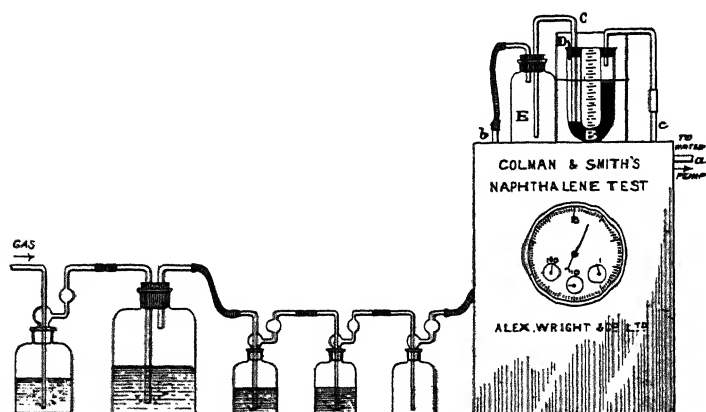


FIG. 38.—COLMAN AND SMITH NAPHTHALENE APPARATUS.

naphthalene picrate is soluble in water. An indiarubber cork fitted with a glass tube at the bottom and having a small hole in the side is then lightly inserted in the bottle, the hole in the tube being just below the bottom of the stopper, and the air in the bottle evacuated with the water pump as completely as possible.

While the pump is still working, the glass tube is drawn up so that the side hole is well within the rubber stopper, the bottle being thus sealed. The bottle is now disconnected from water pump and placed in a water bath, and boiled. The boiling is continued, with occasional shaking of

the bottle, until the solution of naphthalene picrate is quite clear, or until all the free naphthalene has combined with the picric acid forming naphthalene picrate.

When absolutely clear the bottle is removed from water bath and is now allowed to cool. During cooling the bottle must be occasionally shaken to prevent free naphthalene settling out on the side of the bottle. After the bottle has become quite cold, the whole of the naphthalene picrate will crystallise out in fine needle-shaped crystals.

The contents of the bottle are washed into a 250 c.c. flask, the latter filled up to the mark with water, and thoroughly shaken and filtered. The first few c.c. of the filtrate are rejected, as, owing to the filter paper absorbing some of the picric acid, they are weaker than the rest.

100 c.c. of the rest of the filtrate are now taken, and 0.5 c.c. of the lacmoid solution added, and titrated with the standard soda solution until the colour changes to the characteristic green. The 50 c.c. burette supplied with this apparatus is specially graduated on one side, the uncorrected number of grains of naphthalene per 100 cub. ft. is read off direct, but only when the above-mentioned quantities and strengths of solution and volume of gas passed (10 cub. ft.) are adhered to. The reading thus obtained divided by tabular number gives the corrected number of grains of naphthalene per 100 cub. ft.

The meter supplied with this test is so arranged that it can be used in cases where the pressure is not sufficient to drive the gas through the bottles. In this case the water pump is connected with the tube A on the outer meter case and the water turned on. Air or gas is then drawn from the space between the meter and the outer case with which the meter outlet is in open connection, and as the vacuum rises, the mercury level rises in the right-hand limb of the tube B and falls in the left-hand limb, this continuing until the level in the latter falls below the bottom of the tube C. As soon as this happens, air is drawn into the meter case through the side tube D, so that the vacuum

cannot rise higher. The amount of vacuum is regulated by varying the height of the tube c, and must be fixed so that the gas is drawn through the bottles at the desired rate. The bottle E is interposed to catch any globules of mercury which may be carried over with the current of air.

In finding the tabular for correction to N.T.P., the amount of vacuum shown on the mercury gauge must be deducted from the height of the barometer, as this represents the difference between the pressure under which the gas is registered and that of the atmosphere. The facts and reasons of this test are—naphthalene combined with the picric acid forming naphthalene picrate, which has the formula  $C_{10}H_8$ ,  $C_6H_3N_3O_7$ , with free naphthalene, hence the necessity to boil the solution, so that the free naphthalene will be taken up, forming naphthalene picrate, which is an alkali.

It is necessary to remove the ammonia by citric acid, or some other suitable solvent, because this would tend to reduce the acidity of the picric acid, causing an error. The above apparatus is only for use when the strength of the picric acid solution is exactly  $\frac{1}{10}$  normal; this is not always easy to obtain, so the following information will be useful to those who either have not the apparatus or else cannot get the picric acid solution to the desired strength.

Titrate the picric acid solution first with  $\frac{N}{10}$  caustic soda, 1 c.c. of which = 0.0229 gram of picric acid. After passing gas, boiling, &c., titrate again with  $\frac{N}{10}$  caustic soda.

The formula for naphthalene picrate is  $C_{10}H_8$ ,  $C_6H_3N_3O_7$ , *i.e.*, 229 parts of picric acid are united with 128 parts of naphthalene, and therefore the quantity of picric acid

ind  $\times \frac{128}{229} = 0.559$  gives the quantity of naphthalene in the volume of gas used for test.

*Example—*

150 c.c. of picric required 74 c.c. of  $\frac{N}{10}$  soda.

$$\begin{array}{r}
 74 \\
 .0229 \\
 \hline
 666 \\
 148 \\
 148 \\
 \hline
 1.6946 \left\{ \begin{array}{l} \text{gram of picric acid before passing} \\ \text{gas through.} \end{array} \right.
 \end{array}$$

The picric acid afterwards required 67.8 c.c.

Therefore  $67.8 \times .0229 = 1.55262$  gram of picric acid after passing gas.

$$\begin{array}{r}
 1.6946 \\
 1.5526 \\
 \hline
 .1420 \left\{ \begin{array}{l} \text{gram of picric acid lost as naph-} \\ \text{thalene picrate.} \end{array} \right. \\
 .559 \\
 \hline
 12780 \\
 7100 \\
 7100 \\
 \hline
 .0793780 \left\{ \begin{array}{l} \text{gram of naphthalene in quantity} \\ \text{of gas passed.} \end{array} \right.
 \end{array}$$

$.079378 \times 15.43 = 1.2248$  grain in quantity of gas passed.

Quantity of gas at N.T.P. = 10.31 cub. ft.

Therefore  $1.2248 \div 10.31 \times 100 = 11.879$  grains of naphthalene per 100 cub. ft.

**2. Dickenson-Gair's Modification of Colman and Smith Naphthalene Test.**—This method was described by Mr C. J. Dickenson-Gair before the Chemical Industry in December 1905. The author of this process uses acetic acid of a specific gravity of about 1.044.

About 350 c.c. of this acid are taken and put in two bottles (as in Colman test). A small Woulff's bottle containing 150 c.c. of picric acid solution is also added after the acetic acid to act as a catch, and a measured volume of

gas not more than 3 to 6 cub. ft. passed through at the rate of 1 cub. ft. per hour. The ammonia is removed by a suitable solvent. After the experiment is finished the acetic acid and picric acid from the bottles are mixed in a flask, and about 500 c.c. of concentrated picric acid solution added. Pure naphthalene picrate separates out at once in large flocculent masses, which have the advantage of being easily filtered.

After filtering, the naphthalene picrate is dried in vacuo or a warm room and weighed.

This method gives practically identical results as with Colman and Smith method, and the same apparatus is suitable for its use.

There is yet one other method worthy of notice, *i.e.*, one devised by Somerville:—Three glass tubes about 7 in. long and 1 in. in width are fitted up in a similar manner to bottles used in other tests, and about 35 c.c. of 70 per cent. of alcohol are poured into each. The three are closely connected, a measured quantity of gas passed through, and the test disconnected. The contents of the tubes are then washed out and intimately mixed in a flask.

If the gas used is impure, it will be necessary to add concentrated oxalic acid solution until the ammonia is entirely neutralised, as shown by litmus paper. The contents of the flask are now filtered and the filter paper washed with dilute alcohol.

Concentrated picric acid is added in large excess—about 500 c.c. are generally necessary—the liquid agitated and allowed to stand for half an hour. By that time all naphthalene picrate will have separated out, and may be filtered, dried slowly, and weighed. The amount of naphthalene found in the gas varies tremendously, both owing to the class of coal used, temperature of carbonisation, method of taking tar off in the hydraulic main, &c. &c.



The usual amount found is as follows :—

Before condensing, gas	-	-	about 80 grains per 100 cub. ft.		
After condensing, gas	-	-	" 15	"	"
Outlet of purifier	-	-	" 10	"	"
Outlet of works	-	-	" 7	"	"

In this case no naphthalene extractor was at work, the only naphthalene that is extracted being that which was condensed out by condensers, washers, purifiers, &c., but no special effort was made to remove this bye-product.

Many and various have been the methods devised to remove naphthalene from coal-gas. Some have met with success, and others have not come up to or done what was expected of them.

As far as experiments and expert opinion have arrived at the present time, these can be divided into two sections :—

1. Those whose aim and object is to relieve the gas of naphthalene by condensation, and the use of various vapours in the gas to absorb the naphthalene and prevent it from coming down in the crystalline form; and

2. Those whose object is to wash the gas with some solvent of naphthalene, generally a coal-tar oil which absorbs the naphthalene from the gas by bubbling the gas through this solvent, and, when saturated, having fresh oil, &c.

These methods will be considered separately.

In the early days of gas manufacture naphthalene gave a good deal of trouble. We read in Bowditch, "Analysis and Use of Coal-Gas," 1867, of the trouble caused by deposition of naphthalene. Bowditch points out that there is practically an unlimited power or use in condensation, and says it is in the power of most engineers to make considerable difference in their illuminating power how they use or what method they adopt for condensing their gas.

The proper object of condensation is the removal from

the gas of substances produced in the destructive distillation of coal, which for some reason or other are not useful for the purpose of illumination, or which cannot be distributed, but all substances which are useful as illuminants and can be distributed with the gas should be retained. He proposed, in order to accomplish this, that the hydraulic main be kept hot, and that the gas and vapours from this should be passed to a special apparatus kept at a regulated temperature, in which the gas and light hydrocarbons might be separated from the heavier bodies before passing the gas to the purifying plant, so that a larger proportion of the hydrocarbon vapours might be distributed with the gas. This method of condensation he considers would probably lessen the deposition of naphthalene in the gas, &c.

The nearest approach we have to this is the more advanced and scientific method of Dr Colman's "Cyclone" method. In this process the trouble caused by water vapour is greatly diminished by separation of the heavier tar fogs and liquor and gas before it reaches the condenser, therefore increasing the vapour tension of the lighter hydrocarbons and allowing them to exert their solvent action more fully on the naphthalene present. In this process the aim is first to remove any substance that causes the lowering of the vapour tension of the more useful constituents, so that they may have fuller play of their natural tendency to absorb naphthalene, and that later on in the operation when they are condensed the naphthalene will come down dissolved in the condensed matter. There are, one might say, two distinct ideas predominating in the efforts to remove naphthalene, viz., (1) where the object is to utilise the solvent action of the tar either during condensing or before, (2) to use some sort of absorbent for the naphthalene, such as Young and Glover's process, Bell, Colson, &c.

The ideas have been briefly mentioned, but a brief review of some of the more successful methods on these lines may be advantageously given :—

Mr C. E. Botley, Engineer of Hastings Gas Works, employs what might be called an intermediary process, the oil being introduced in the form of minute atoms, which are carried along with the moving gas, and apparently hold up the naphthalene. Anyhow it was definitely proved at Hastings that this fog was carried two miles or more from the works, and if these atoms of oil could be carried to the end of the district, Mr Botley claims that naphthalene troubles would cease.

The next idea of mark is C. Carpenter's reversible condensers (*Journal of Gas Lighting*, 19th Nov. 1901). Some six years prior to this paper Mr C. Carpenter brought forward the idea for the removal of the surplus naphthalene contained in crude coal-gas by means of a reversible condenser, in which the deposit naphthalene thrown down upon the water-cooled walls was dissolved by the hot tarry vapours of the foul main gas, and run off through suitable seals into the tar well. The condenser used was an old one; it possessed, however, two qualifications which were considered important, *i.e.*, it was vertical and water-cooled. In order to effect the reversing of the current of gas, and make this flow from left to right, or right to left, as desired, a special four-way valve was fitted up. The experiments on this apparatus proved successful, but apparatus too small, and Mr Carpenter adopted the following:—The new condenser had capacity of 2 million cub. ft. per diem; it was decided to adhere to water as the cooling medium in order to render the reduction of temperature as far as possible independent of atmospheric conditions. The vertical type was also adopted, as a result of the satisfactory working of the experimental apparatus, the facility which the vertical afforded for the "buttery" naphthalene to slide down to the seal pots, and the further advantage that the drainage of these more or less liquid products was independent of the direction in which the gas was flowing in the apparatus.

The following is a description of the apparatus used:—It consists of two rows of seven steel pipes,

27 feet long, formed into a horseshoe by a bridge piece connection at one end. Within each of these pipes are sixteen 2-in. tubes, running from top to bottom, screwed into the bottom diaphragm separating the water and gas spaces. The expansion of these tubes under the extremes of temperature worked is about  $\frac{1}{4}$  inch, and the top ends, therefore, after reduction in size to save friction, and also to distribute the flow of water, work through stuffing boxes carried in the upper diaphragm. The working gas valves consist of two pairs of ordinary slide valves, each pair geared together—one open, one shut—by means of a shaft which carries two pinions operating the racks of the valve in opposite directions. Two pairs of water valves are also provided, but these work independently. Each pair of the vertical condensing pipes drain into a double seal pot. The consistency of the dissolved naphthalene is such that, in order to maintain it sufficiently fluid, hot liquor must be continually running through the pipe, which forms the centre of the seal, and this precaution has been found essential even during the heat of summer. Valves are provided to each seal so as to give access thereto in case of stoppage, the condenser being worked under pressure on the exhaust outlet.

The condenser had been at work some six months, and since that time hardly a flake of naphthalene crystal had been seen on the works or outlet main. The meter overflow, which used to choke every few days, had remained clear, and so on throughout the plant.

Naphthalene test on the outlet gas at station meter showed from  $1\frac{1}{2}$  to  $3\frac{1}{2}$  grains naphthalene per 100 cub. ft.

The next paper on the subject is by Dr H. G. Colman (appearing in the *Journal of Gas Lighting* for 3rd June 1902). Attention is confined in this paper to the separation of the tar vapours from the gas, without consideration of the condensation of the aqueous vapour, beyond stating the view that its influence on the removal of naphthalene, and the subsequent illuminating power of the gas, is only

of subsidiary importance, as water does not dissolve, and is not dissolved by the tar constituents to any considerable extent, and its separation therefore takes place practically as it would if no tarry vapours were present. The aim of condensation may be defined as the reduction of the gas to the atmospheric temperature, and the simultaneous removal, as completely as possible, of all substances which are not permanent gases at that temperature, with the exception of the lowest boiling hydrocarbons (practically benzene and toluene). Of these latter, it is desired to keep in the gas the maximum quantity it is capable of retaining at the lowest temperature to which it may be exposed during distillation.

The idea in this paper was to separate the removal of the heavy tar as soon as possible, and by keeping the gas in contact with the light oil or tar fog containing minute globules of light oil as long as possible.

The chief action is attributed to the action of the tar fog; the conveying of the gas through a great length of foul main at a slow speed is favourable, inasmuch as it allows the condensation of much heavy tar which would otherwise have reached the condensers, but the continuance in the condensers of slow cooling is not advantageous, inasmuch as owing to the low velocity of the gas, and the longer time it is passing through the condensers, the light oil fog is largely deposited and removed from further action on the gas before it reaches the cold end of the condenser, where its solvent power for naphthalene is greatest.

Hence, if these views are correct, it follows that, while slow condensation up to a certain point is desirable, the rate of cooling at the later stages must be sufficiently rapid to carry the light oil fog formed at the inlet to outlet of condensers. It is most important that the heavy tars have been previously removed. If this is not the case quick condensation will not effect the sufficient removal of naphthalene, but will also bring about the absorption of benzene vapour, which would otherwise have remained in the gas,

thereby decreasing the illuminating power. The mere lowering of the temperature of the gas from 75 to 60 degrees in the absence of solvents cannot, however, be expected to effect much in the naphthalene, inasmuch as the quantity present in the gas which has only been cooled to 75 degrees is less in many cases than is necessary to saturate it at 60 degrees. The points to be considered are (1) the complete removal of the heavy tar fog before the condenser; and (2) ensuring the presence of a sufficient quantity of light oil fog in the colder portions of the condenser. The first point, the object is in part obtained by employing long foul mains of ample size, but even when this is done, the gas at the inlet of condenser still contains a fairly dense fog. With the view of replacing these miles of main Dr Colman employed a centrifugal separator modelled on the lines of the well-known "Cyclone" dust collector. The gas enters through a pipe of oblong section at a tangent, thus bringing about a rapid circular motion of the gas in the cone. Any solid or liquid particles are driven by centrifugal force to the circumference, and fall to the bottom, where they are taken off direct to a seal pot. The outlet is at the centre of the cone, so that the gas is drawn off at the point where it is freed from suspended particles. The velocity of the gas is controlled by a flap-valve at the inlet of cone.

The next process is the washing of the gas by solvents, which absorb the naphthalene in the gas. There are two or three different ideas as to the most suitable solvent for this purpose. Messrs Young and Glover took out a patent in 1897 for washing gas with oil for the removal of naphthalene, and were undoubtedly the pioneers who first put this method before the gas industry. Since then both Mr Coulson, of Leicester, and Mr F. Bell, of Derby, have obtained success or freedom from naphthalene troubles by washing gas with some solvent oil.

In July 1904, Mr Coulson, of Leicester, took out a patent on these lines:—

**My invention consists in an improved solvent liquid**

capable of removing naphthalene with certainty from illuminating gas obtained by distillation, and in a method of preparing this liquid.

The raw material employed is coal-tar, or in general any tar. Such tar is distilled as usual, and that portion that comes over below 270 degrees Cent. is then further distilled to isolate the mixed oils, chiefly those having a boiling point between 170 and 215 degrees Cent., and which contain only a small quantity of naphthalene.

The oil thus obtained is capable of extracting naphthalene from the gas rapidly and with certainty. The density of the oil thus prepared is from 994 to 998, or even a little heavier.

The washing of the gas is best carried out in some form of washer.

Mr Coulson found that it required 0.08 gallon of the solvent per 1,000 cub. ft. of gas.

This process has met with complete success in Leicester, completely removing all troubles caused by naphthalene deposits.

Mr Bell's process is on similar lines, but his is a dual process, as he first washes his hot gas with hot tar, and then with heavy naphtha. He also claims complete success.

These various processes are given as an indication of the trend of opinion on how to remove the naphthalene nuisance, but up to the present there does not appear to be any universal solvent or method that will cure naphthalene troubles in any town, for it is apparent that the process that meets with success in one place may fail altogether in another district.

# CHAPTER X.

## THE ANALYSIS OF FIRE-BRICKS AND FIRE-CLAY.

FIRE-CLAYS are clays which are capable of standing a very high temperature without fusing. Such clays are said to be refractory.

The composition of fire-clay varies, however ; the following Table gives the average analyses:—

COMPONENTS.	1	2	3	4	5	6	7
Silica . . .	62.35	56.42	67.50	73.27	71.97	96.25	65.10
Alumina . . .	18.47	26.35	22.39	21.62	24.20	1.00	22.22
Ferric oxide . . .	4.77	1.33	6.37	4.24	1.80	1.15	1.92
Lime . . .	trace	0.60	1.55	0.58	0.224	1.07	0.14
Oxide of man- ganese . . .	...	...	0.44	trace	0.76	Nil.	...
Magnesia . . .	1.36	0.55	trace	trace	0.096	...	0.18
Sodium . . .	...	...	1.30	0.29	0.950	0.53	...
Potash . . .	2.47	0.48					
Organic matter	5.22	...	...	...	...	...	0.58
Combined water . . .		10.95	...	...	...	...	7.10
Moisture . . .	4.15	2.80	...	...	...	...	2.18
Titanic acid . . .	1.10	1.15	...	...	...	...	...
Fire resistant . . .	...	...	89.89	94.89	96.17	97.25	...
Fusible con- stituent . . .	...	...	10.16	5.11	3.83	2.75	...
	99.89	100.63	100.05	100.00	100.00	100.00	99.42

1. Derbyshire fire-clay (Riley). 2. Glenboig fire-clay (Riley). 3. Fire-brick, bad quality. 4. Retort made in Stourbridge not satisfactory owing to iron. 5. A fire-brick of satisfactory quality. 6. Silica brick of satisfactory quality. 7. Stourbridge clay.



A good refractory fire-clay will contain nearly pure hydrated silicate of alumina. The more alumina that a fire-clay contains in proportion to the silica, the more refractory will be that clay.

On a careful observation of these analyses, it will be seen that the fire-resisting constituents are silica, alumina, and that any heavy proportion of oxide of iron, or alkalies, act as a flux and cause fusion; the clay is no longer refractory.

**The Method of Analysis.**—A quantity of the substance (fire-clay or fire-brick) is reduced to an impalpable powder in an agate mortar. It is absolutely necessary, in order to ensure the complete decomposition of the silicate, that the powder should be so fine that there should be no grittiness to the touch when it is rubbed between the thumb and finger. The whole sample when thus ground should pass through a sieve of fine muslin.

About 5 grams of the sample are dried in a platinum crucible or dish at a temperature of 100 degrees Cent. in a water bath until the weight is constant; the loss in weight gives the moisture.

In the case of a clay it is ignited at first gently, and then placed in the combustion furnace for a tolerably long time. The loss in weight gives the combined water, organic matter, and volatile constituents of the clay, if such are present.

**Silica** ( $\text{SiO}_2$ ).—2 grams of the finely powdered sample (dried) are weighed out in a fairly large platinum crucible, and about six times its weight of fusion mixture added (sodium and potassium carbonate mixed in molecular proportions). The whole is intimately mixed by means of a smooth rounded glass rod. The mixture should only half fill the crucible. The lid is then placed on the crucible, and the latter is then heated gently over a Bunsen flame, the temperature is gradually increased, great care

being taken that no loss ensues by the frothing due to the evolution of carbon dioxide. When the mass is fused, it is then heated by means of the blow-pipe, the heating is continued until all effervescence ceases, and the contents of the crucible are in a state of quiet fusion. When this is finished the crucible is allowed to cool down, and when cold it is placed on its side in a beaker with about 100 c.c. of cold water, great care being taken that no impurities are conveyed into the solution upon the outside of the crucible. The beaker is heated on a sand bath until the "fusion" is detached from the crucible. Hydrochloric acid is now gently added in small quantities at a time (the watch glass being replaced between each addition), until effervescence ceases, and no further precipitate of gelatinous mass takes place. The crucible and lid are now withdrawn with platinum-tipped tongs and rinsed into beaker.

The mixture is now transferred to a platinum dish and evaporated to dryness upon a steam bath, the gelatinous mass being stirred at frequent intervals with a round glass rod to prevent the formation of lumps. In order to expel the last trace of HCl, the dish is transferred to an air bath and heated to about 160 degrees Cent. for half an hour. The residue is then moistened with a little HCl, and is then heated on a water bath for about half an hour, more HCl being added as evaporation takes place. Hot water is added and the silica is filtered off, and is washed free from dissolved chlorides.

The precipitated silica is dried in the oven; it is then ignited apart from the filter, the precipitate being transferred to the platinum crucible very cautiously, as, since it consists of a very light powder, it is easily blown away. The covered crucible is at first heated very cautiously, and afterwards to a red heat, and weighed until constant.

**Alumina ( $\text{Al}_2\text{O}_3$ ) and Ferric Oxide ( $\text{Fe}_2\text{O}_3$ ).—The**

iron and alumina are precipitated together in the form of hydroxide by the addition of ammonium chloride and ammonia to the filtrate from the silica. They are separated as under.

The precipitate is washed and dissolved upon the filter with hot dilute HCl, and the solution allowed to flow into a porcelain or platinum dish, which contains about 50 c. c. of pure strong KOH solution. The filter paper is washed with a small quantity of distilled water, and these washings are allowed to run into dish.

The iron will be precipitated as ferric hydrate, while the hydrate of aluminium will remain in solution. The precipitated iron is filtered off and redissolved in HCl, and reprecipitated by  $\text{NH}_4\text{OH}$  to free it from potash. It is then washed, dried, and ignited apart from the filter at a red heat, and weighed as  $\text{Fe}_2\text{O}_3$ .

The solution of aluminium hydrate in the potassium hydrate solution is treated with a slight excess of HCl, and then with a slight excess of  $\text{NH}_4\text{OH}$ . The precipitate is then filtered off, washed and dried, ignited and weighed as  $\text{Al}_2\text{O}_3$ .

**Calcium.**—If the filtrate and washing from the iron and alumina precipitate is large, evaporate down to about 150 c. c., add a little  $\text{NH}_4\text{OH}$ , and then a slight excess of ammonium oxalate, filter, ignite, and weigh the precipitate. From the result thus obtained, the percentage of calcium oxide,  $\text{CaO}$ , is calculated.

**Magnesium.**—The filtrate and washing from the calcium oxalate precipitate is evaporated to dryness, ignite the residue, and treat it with a little strong HCl, add water, and filter if necessary. To the filtrate add  $\text{NH}_4\text{OH}$  in moderate excess, and then an excess of a solution of hydrogen disodium phosphate. Allow the liquid to stand for a few hours, as the precipitate is a crystalline compound. Its formation is accelerated by vigorously shaking in a

stoppered bottle; filter off, wash precipitate with dilute ammonium hydrate solution, then ignite it, and weigh the Mg as  $Mg_2P_2O_7$ .

**Potassium and Sodium.**—As sodium and potassium carbonates have been employed in "opening up" the silicate, it is obvious that the alkali metals cannot be estimated in a solution so obtained.

A separate portion of the substance must accordingly be used for their determination.

Weigh out 2 grams of the finely powdered substance into a platinum crucible. Well mix it with about six times its weight of pure calcium carbonate, and its own weight of pure ammonium chloride. The platinum crucible is placed in a clay crucible containing a little lime or calcined magnesia at the bottom and round the sides. The whole is now placed in a furnace, and maintained at a bright red heat. This heat is maintained for one hour. The crucible is now withdrawn, allowed to cool, then place the platinum crucible and contents in hot water in a covered platinum or porcelain crucible or dish, and boil for a time. The crucible is now withdrawn and rinsed, and the liquid is filtered, the residue being well washed. The solution now contains the alkali metals in the form of chlorides, is freed from any lime salts which have dissolved by the addition of ammonia, ammonium carbonate, and ammonium oxalate. The precipitate is filtered off, evaporate filtrate to dryness with a few drops of HCl in a weighed platinum dish; finally raise to redness. The residue is now gently ignited and weighed until the weight is constant. The weight thus obtained gives the combined weight of potassium and sodium chlorides.

In order to estimate the relative proportions of the potassium and sodium chlorides, the residue is dissolved in a very small quantity of water, and the potassium precipitated by the addition of an excess of platinic chloride. Adding a few drops of hydrochloric acid the

mixture is now evaporated on a steam bath until it becomes semi-solid.

Alcohol is now poured on the mass, and the liquid gently shaken round in the dish, so as to well mix the contents; the precipitate is allowed to settle completely, and the liquid poured off through a tared filter paper. The precipitate in the crucible is washed with alcohol two or three times; the undissolved precipitate is now transferred to the filter by means of a small wash-bottle filled with alcohol. The precipitate is washed with alcohol until the washings are no longer coloured. The filter is then withdrawn from the funnel, folded, and placed between a pair of watch glasses, and dried in the oven at 100 degrees Cent., and weighed as  $2\text{KClPtCl}_4$ . On deducting the weight of potassium chloride so obtained from the weight due to the mixed chlorides, the proportion of sodium chloride is found.

#### **Specific Gravity, Volume Weight, and Porosity.—**

In the *Society of Chemical Industry Journal*, 15th February 1906, p. 102, Dr E. A. Wagstaffe, in a paper entitled "Chemical and Physical Valuations of some Clays and Shales for Brickmaking, chiefly from East Cheshire," gives the following:—

The weight of the briquette having been ascertained, the briquette was soaked in water for twenty-four hours, taken out, and the surface water removed with a dry cloth. The increase in weight gives the amount of water absorbed by the briquette, *i.e.*, the amount that fills the pore spaces. The volume of the briquette is determined by displacement of water. The volume may also be determined by measurement, provided the briquette has not suffered any unequal contraction, whereby the shape has become distorted. Then if the weight of the briquette in grams is  $w$ , and the increase in weight due to absorption of water is  $t$ , and the volume in c.c. displaced when water saturated is  $v$ ,

$$\text{the specific gravity} = \frac{\tau e'}{(e' - t)},$$

$$\text{the volume weight} = \frac{\tau e'}{e'},$$

$$\text{porosity} = \frac{100 \times t}{e'}.$$

**Analysis of Weldon Mud.**—This material for gas purification was brought before the gas world by J. J. Hood and A. Gordon Salamon in a paper entitled "The Application of Weldon Mud to Gas Purification," read before the Institute of Gas Engineers, and published in the *Transactions* of that Institute for 1893.

In this paper the authors say: The material is that which is known to the alkali industry as Weldon mud, a compound consisting essentially of hydrate oxide of manganese.

It is affirmed, as a great recommendation of Weldon mud, that the spent material can be regenerated upon an economical basis for further use in gas purification. Theoretically there should in this process be no loss of the manganese constituents of the Weldon mud.

It is worked through a cycle, viz., manufactured, next employed in the removal of sulphur, and when sufficiently saturated, submitted to regeneration.

During this latter process it parts with all its sulphur, and as a result of the regeneration it is restored to its original condition, and is ready for further use by the gas engineer. Its regeneration being economical, the authors assert that it will be found, when worked under the best conditions, to compare favourably in price with other gas-purifying materials.

The Weldon mud was found to be a sharp and powerful absorbent of sulphuretted hydrogen, and that the sulphide of manganese resulting from such absorption could be revived *in situ* by the admission of a regulated sufficiency of air.

It was found that Weldon mud required about 1.0 per cent. of atmospheric air for revivification *in situ*, and that when this quantity was admitted it remained active for some considerable period. If the Weldon mud be in a position where condensation is a matter of certainty, it will, by virtue of its physical structure, absorb the water thus condensed, and when saturated may have to be taken out in a wet and sloppy condition.

If the mud thus removed be spread in thin layers, and freely exposed to the action of the air it will dry, and can be broken, &c., and will then be active for further sulphuretted hydrogen absorption.

The analysis of a sample of Weldon mud works out as follows:—

Water	-	-	-	40.60 per cent.
Manganese dioxide	-	-	-	29.80 „
Calcium carbonate	-	-	-	10.60 „
Calcium chloride	-	-	-	3.01 „
Calcium sulphate	-	-	-	2.64 „
Calcium hydrate	-	-	-	3.01 „
Loss on ignition	-	-	-	9.80 „
Fe <sub>2</sub> O <sub>3</sub> , MnCl <sub>2</sub> , &c.	-	-	-	0.54 „
				<hr/>
				100.00 „

Great care must be given in taking a representative sample of Weldon mud; it should fill the bottle completely, be well corked and sealed, if not, owing to various changes in temperature, small globules of water will separate out on to the sides of the bottle. It is only usual to analyse the sample for the amount of water, and for the manganese dioxide. The samples usually contain from 40 to 46 per cent. of water, and from 25 to 32 per cent. of manganese dioxide. The moisture is estimated in the usual manner by drying in a water bath at 212 degrees Fahr. until a constant weight is obtained, twenty-four hours being generally sufficient for this purpose.

For the estimation of the manganese dioxide the available oxygen is the oxygen which can be made use of for

oxidising purposes, when the ore is decomposed by an acid. The method used is where the evolved oxygen is used in oxidising a ferrous salt; therefore, if a known quantity of Weldon mud be dissolved in sulphuric acid, in the presence of a known quantity of ferrous salt in excess, and if the amount of the ferrous salt which remains unoxidised be then determined by means of a decinormal permanganate of potash solution, the quantity of iron oxidised, and therefore the amount of manganese dioxide, can be calculated.

The  $\frac{N}{10}$  permanganate solution is made as usual, and the iron solution is prepared as follows:—100 grams of pure, dry, clean crystallised ferrous sulphate are dissolved in distilled water, to which is added 100 c.c. of pure concentrated sulphuric acid, and the total made up to 1,000 c.c. at 60 degrees Fahr. with distilled water. The exact strength of this solution is ascertained by titrating 20 c.c. with the decinormal permanganate. If the ferrous sulphate is pure and unoxidised it will require 72 c.c. of the decinormal permanganate to oxidise the 20 c.c. The exact amount of permanganate required is noted; this must be carried out for every test, because the solution will oxidise even when kept in very closely stoppered bottles.

0.5 gram of Weldon mud is now weighed out, washed into a small beaker, and 20 c.c. of the iron solution added. The whole is now boiled until all the Weldon mud is dissolved, being aided if necessary by breaking the lumps with a glass rod (flattened at one end) against the bottom of the beaker. When the mud is completely dissolved the solution is titrated with the decinormal permanganate, and the volume of the latter required for oxidation noted.

*Example—*

20 c.c. of ferrous sulphate solution required 71.6 c.c.  $\frac{N}{10}$  permanganate solution for oxidation.

0.5 gram Weldon mud dissolved in 20 c.c. of ferrous



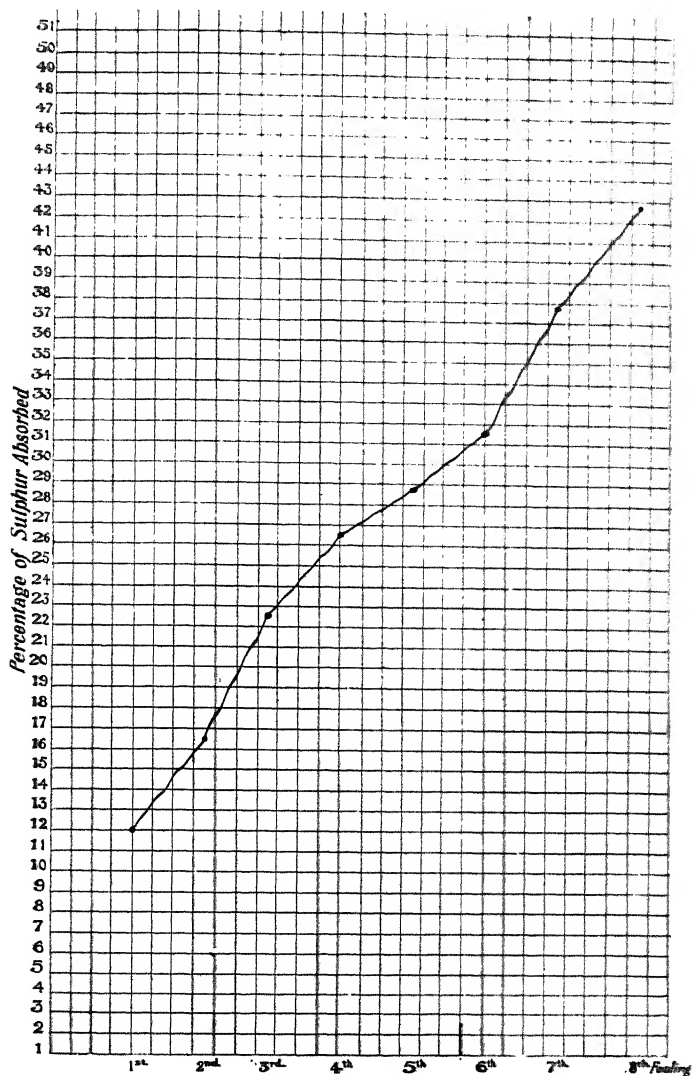


FIG. 39.—CHART SHOWING SULPHUR (DRY BASIS) ABSORBED BY WELDON MUD.

sulphate solution required 43.10 c.c. of decinormal permanganate solution.

Therefore  $71.6 - 43.1 = 28.50$  c.c.  $\frac{N}{10}$  permanganate.

Therefore, as 1 c.c. of decinormal permanganate is equivalent to 0.00435 gram of manganese dioxide, then the 0.50 gram Weldon contains  $38.5 \times 0.00435 \times 200 = 24.795$  per cent. of manganese dioxide.

Sometimes there is a slight difference in the analyses done by various people; if the analyses are calculated on the dry basis it will be found that they generally agree. It is usual to do at least two samples of Weldon mud, and they must agree to 0.20 per cent., if not, a third must be done.

If the affinity of a sample of Weldon mud for sulphuretted hydrogen is required, it is carried out in a similar manner as that described under oxide of iron.

The chart on previous page shows the percentage of sulphur which is usually absorbed by Weldon mud without the addition of oxygen (in the form of air) for revivification *in situ*, the revivification being carried out separately for each fouling.

**Analysis of Spent Oxide.**—This method is based on the fact that free sulphur is dissolved by bisulphide of carbon. This bisulphide is evaporated off, and the sulphur is left behind in the solid form, in which state it may be weighed.

It is necessary to remember that carbon bisulphide will only dissolve the free sulphur, and it is advisable to spread some of the sample out to the air for complete revivification before proceeding to extract the sulphur.

Carbon bisulphide only imperfectly dissolves sulphur in the presence of water, so that the sample must be dried at 212 degrees Fahr. first. The apparatus employed is shown in Fig. 40, and was designed by Mr A. Stephenson of the Gas Purification Company.

50 grains of spent oxide are weighed out on a tared watch glass, and dried at 212 degrees Fahr. for moisture. This is weighed until the weight is constant and gives the moisture in the sample. Another 50 grains of the material which was placed out for complete revivification is dried in the oven. This does not give the true moisture, as it may have dried whilst laying out. This sample is carefully placed in tube A on the top of a layer of cotton wool. After putting in the dried oxide more cotton wool is put in on top. Bisulphide of carbon is blown from bottle B into test-tube A on top of the spent oxide. The  $\text{CS}_2$  gradually percolates

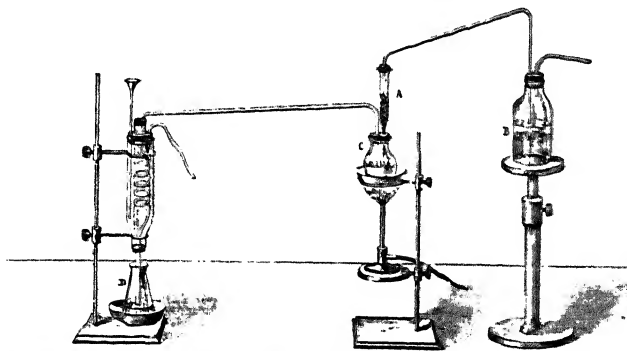


FIG. 40.—APPARATUS FOR ESTIMATION OF SULPHUR IN SPENT OXIDE OR WELDON MUD.

through the material, dissolving the sulphur in its course, finding its way into flask C (which has been previously dried and weighed). The flask C is placed in a copper water bath which is kept boiling by the Bunsen burner. The  $\text{CS}_2$  is driven off and is condensed in its passage through the condenser, and is recovered in the liquid state in flask D. The recovered  $\text{CS}_2$  can be used over and over again. The sulphur remains behind in flask C. The oxide is well washed with  $\text{CS}_2$  until free from sulphur, and when the sulphur is solid in the flask C it is disconnected and put into a water bath for three or four hours

to eliminate the last traces of  $\text{CS}_2$ . Place in desiccator and weigh when cold. The increase in weight  $\times 2 =$  percentage of sulphur on wet basis.

It should be remembered that  $\text{CS}_2$  is very inflammable, and in the gaseous state, when mixed with certain percentage of air, highly explosive. The bisulphide should always be covered with water.

Another method for extraction of sulphur is by the ordinary Soxhlet apparatus. A most useful adaptation of this is shown in Fig. 41. The only other compound required to be estimated in spent oxide is the total ferrocyanide. This is not usually extracted in the oxide in this country, but sometimes it is. The method is as follows:—

A weighed portion, say 10 grams, of the spent oxide is well powdered in a mortar, and is digested with a strong solution of caustic soda boiled, and the solution filtered off. The total ferrocyanide can be estimated in many manners—(1) by precipitating as Prussian blue as given in cyanogen analysis; (2) by zinc sulphate; or by the follow-

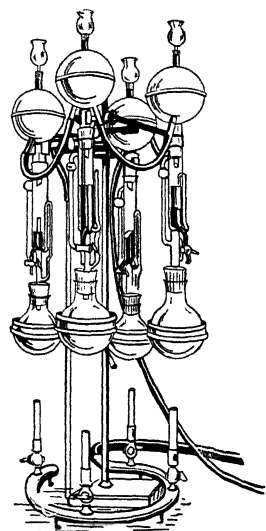


FIG. 41. — SOXHLET APPARATUS FOR THE ESTIMATION OF SULPHUR IN SPENT OXIDE OR WELDON MUD.

ing method:—The caustic soda solution is evaporated to dryness, with sulphuric acid in excess. The residue is dissolved in dilute sulphuric acid, filtered, and the iron in the filtrate reduced by zinc. Titrate with  $\frac{N}{10}$  potassium dichromate.

From the amount of iron so found the amount of Prussian blue can be calculated.

## CHAPTER XI.

### *PHOTOMETRY AND GAS TESTING.*

THE art of photometry is the art by which the ratio between the amount of light emitted by two sources is ascertained.

The first law is "Kepler's Law of Inverse Squares," which is that "the quantity of light falling on a given surface varies inversely as the square of the distance from the source."

The second law is "Lambert's Cosine Law," which, however, is only approximately true, and is that "the intensity of illumination which is received obliquely, is proportional to the cosine of the angle which the luminous rays make with the normal to the illuminated surface."

The third law is the "Generalised Photometrical Law" which is that  $e = \frac{I \cos. \theta}{d^2}$ , where  $e$  is the light falling on a given surface,  $I$  the intensity of the source,  $\theta$  the angle of incidence of luminous ray, and  $d$  the distance of the source from the screen.

Now with these three rules, which form the basis of all photometrical works, no further study of physics is absolutely necessary for the comprehensive study of photometry.

In the early days of photometry the standard of light was the sperm candle of six to the pound, each burning 120 grains per hour. Few candles burn at this exact rate, and in practical photometry a correction is made for the

amount of sperm consumed, either more or less than this amount.

Numerous other standards have been proposed during the last few years, and the official standard of light is now Mr Vernon Harcourt's 10-candle pentane standard lamp.

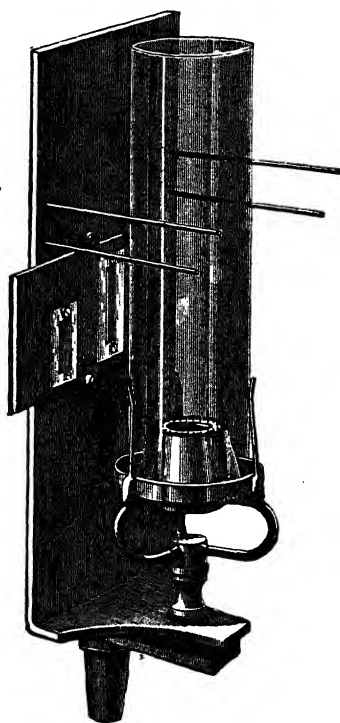


FIG. 42.—METHVEN SCREEN.

This 10-candle pentane lamp is one in which air is saturated with pentane vapour, the air gas so formed descending by its gravity to a steatite ring burner. The flame is drawn into a definite form, and the top of it is hidden from view by a long brass tube, in which the air is warmed by the chimney, and so tends to rise. This creates a current, which, descending through another tube, supplies air to the centre of the steatite ring burner. No glass chimney is required, and no exterior means have to be employed to drive the pentane vapour through the burner. (Complete particulars are given in Appendix A.)

The next standard of light substituted for the old sperm candle is the Methven screen, modified forms of which are used where officialism does not stipulate for the use of the Harcourt standard. The invention originally appeared about 1878, and is the invention of Mr John Methven of the Gas Light and Coke Co. In a communication to the meeting of the Gas Institute in June 1882, Mr Methven described a modified form

of this standard. This consisted of a standard Argand burner consuming coal-gas enriched by the vapour of pentane. A screen allowing only the light from a small portion of the flame to pass through was used.

The flame of the standard Argand used was regulated to a height of  $2\frac{1}{2}$  in., and the dimensions of the slot were altered from his original form to suit the shorter and more luminous flame. The dimensions given by Mr Methven are in the above paper, being about 15 mm. high and about 8 mm. wide, and the light was to be taken from a portion 24 mm. above the burner. Heisch & Hartley issued in 1883 a very favourable report on the Methven

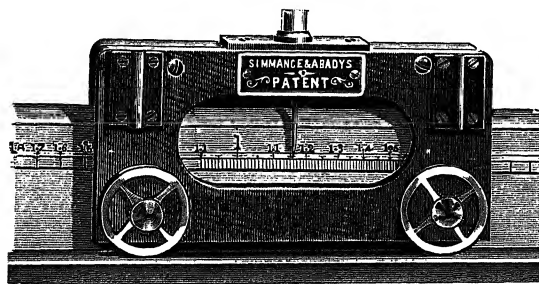


FIG. 43.—GRADUATED BAR WITH CARRIAGE.

screen as a method of obtaining a convenient standard light. The height of the flame should not deviate from the normal.

As will be seen from the illustration, the Methven screen has two bars for adjusting the height of the flame, and two slots. The lower bar and smaller slot are for use on carburetted coal-gas, and the higher bar and larger slot for use on uncarburetted coal-gas.

The carburettor, which must be perfectly air-tight, consists of a metal box with inlet and outlet pipes, both provided with taps; there is also a by-pass between the inlet and outlet pipes, likewise furnished with a tap, so that the gas can be supplied to the burner either carburetted or uncarburetted.

**Photometrical Testing.**—The photometrical room should be of convenient dimensions. It should be well ventilated and free from draughts, not subject to vibrations, and should be maintained as near as possible to a temperature of 60 degrees Fahr. as circumstances will permit. The walls of the room should be coloured dull black, but need not necessarily be black all over, but should preferably be of a dull colour.

The apparatus consists generally of what is known as the Letheby-Bunsen Photometer (except where the Referee's apparatus is used, see Appendix A.).

The complete apparatus consists of a candle balance or some other standard, Bunsen disc box or Simmance-Abady "Flicker" attachment, Argand burner for testing gas under consideration, meter, governor, &c., as shown in figure.

When using the candle balance, the Gas Referees assume that the illuminating power of a candle varies strictly as the rate of consumption of the sperm for variations of not more than 6 grains per hour above or below the normal rate, *i.e.*, if the rate of consumption of a candle exceeds 126 grains, or falls short of 114 grains per hour, any test made under these circumstances must be discarded.

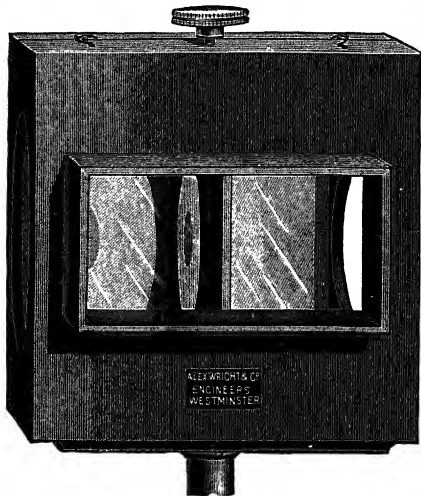


FIG. 44.—BUNSEN REVERSIBLE DISC BOX WITH MIRRORS.



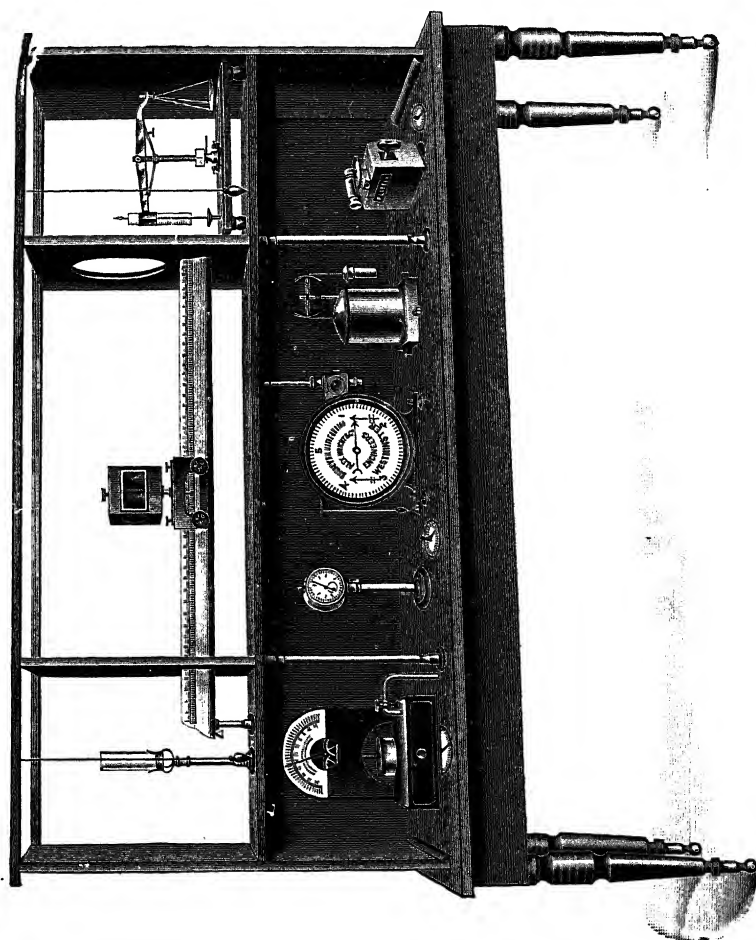


FIG. 45.—6-INCH LETHEBY-BUNSEN PHOTOMETER.

**The Candle Balance.**—The method of working the candle balance is as follows:—A candle is cut in half and fixed in the clips provided; this is best done by laying the candle on a clean level surface, and rolling it under the edge of a sharp knife. Cut away half an inch of sperm from the top of the lower half, and the same from the bottom half of the other, reduce the wicks to half an inch in length. The candles are now ready for burning, and

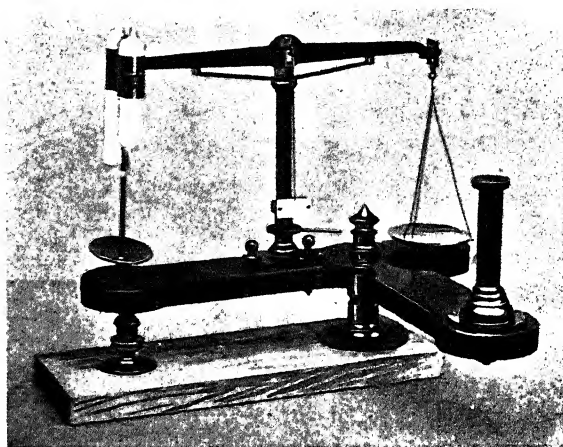


FIG. 46.—CANDLE BALANCE.

should burn at least fifteen minutes before testing. The wicks of the candles should bend away from each other.

Put weights in candle pan of balance until candles are slightly heavier than counterbalance. As the burning of candles makes them lighter than the counterbalance, the pointer will move over indicator, and as it passes the zero mark on indicator start the test clock. Gently lower beam until both pans are at rest, and then add the 40 grains weight to the candle end, and gently lift beam so that balance is once more in action.

Make readings on bar by adjusting disc box until the grease spot is equally indistinct on either side, and note down reading. Ten observations are made, one at the beginning of each minute.

*Example—*

1st minute	-	-	-	-	-	-	8.2
2nd "	-	-	-	-	-	-	8.3
3rd "	-	-	-	-	-	-	8.4
4th "	-	-	-	-	-	-	8.5
5th "	-	-	-	-	-	-	8.0
6th "	-	-	-	-	-	-	8.2
7th "	-	-	-	-	-	-	8.4
8th "	-	-	-	-	-	-	8.3
9th "	-	-	-	-	-	-	8.2
10th "	-	-	-	-	-	-	8.2

---

82.7

The disc is reversed at end of 5th minute to balance any possible inequality in the two sides.

Divide 82.7 by 10 = 8.27.

After the tenth reading watch candles very closely, and directly the pointer begins to move, be prepared to stop the clock directly the pointer passes zero mark on indicator (showing that 40 grains have been consumed), stop clock and note time, say 9 minutes 45 seconds.

In this test the gas under observation is always burnt at 5 cub. ft. per hour, so that it only requires an occasional glance to ascertain that this rate is constant, which is an easy matter; but say the consumption is a little fast, viz., 5.1 cub. ft., we now have the following data :—

1. The time taken to burn 40 grains	-	-	9' 45"
2. Average reading of candle power	-	-	8.27
3. Gas consumption	-	-	5.10

The working of the result is :—

$$\begin{array}{r}
 8.27 \\
 \underline{2} \\
 16.54 \\
 \underline{5} \\
 5.1)8270(16.21 \text{ candles at 5 cub. ft. per hour.} \\
 \underline{51} \\
 .317 \\
 \underline{.306} \\
 .110 \\
 \underline{.102} \\
 .80
 \end{array}$$

Corrected average for gas = 16.21

Time due to 40 grains consumption in seconds = 600

9726.00

Time actually occupied 9' 45" = 585 seconds.

Then—

$$\begin{array}{r}
 585)9726.00(16.62 \\
 \underline{585} \\
 .3876 \\
 \underline{.3510} \\
 .3660 \\
 \underline{.3510} \\
 .1500
 \end{array}$$

16.62 ÷ Tabular number, correction for temperature and pressure = 1.036.

1036)16.62(16.04 equals corrected candle power.

$$\begin{array}{r}
 1036 \\
 \underline{1036} \\
 .6260 \\
 \underline{.6216} \\
 .4400
 \end{array}$$

Therefore the correct illuminating power of the gas tested is 16.04 candles at the 5 cub. ft. per hour rate.

For using the Methven screen or Harcourt standard fitted to this type of photometer the method can either be on the 5 cub. ft. per hour rate, or on the 16-candle basis.

*The 5 cub. ft. per hour rate.*—The gas is burnt at the rate of 5 cub. ft. per hour, and readings are made on bar by adjusting disc box as before, and in the case of Methven screen (being a 2-candle standard) multiply readings by 2 and correct for N.T.P.

*Sixteen-Candle Basis.*—The disc box is set at the figure 8, and the gas is regulated so that the grease spot is equally indistinct on either side, then the minute clock is started, and the rate of consumption taken.

*Example—*

Disc set at 8.0, rate of consumption 5.1 ;  
then—  
5.1)8.000(15.68 candles.  
51  
—

and  $15.68 \div 1.012$  (Tab. No.) = 15.49 corrected candle power.

The complete method of gas testing as required by the Gas Referees is given in the Appendix A.

**The "Flicker" Photometer.**—Patent No. 4,693, 28th February 1903, by J. F. Simmance and J. Abady.

In the specification it is set out that careful investigation and experiments have proved that the physiological causes of the little known optical phenomena described herein have been hitherto misunderstood, and attempts to utilise them in photometry have in consequence been abortive. The blurring, flickering, or throbbing effect produced upon the eye by the rapid alternation of rays from the light under test and a comparison light (each of a distinct tint and intensity) is caused by the anxiety of the nerves controlling the dilation and contraction of the pupil to fulfil their office, whilst the effort to do so is frustrated by the rapid changing. The relief afforded by the arrival

at equal intensities of the two lights signals unmistakably the point when such equality has been reached.

To arrive at the comparative intensities of light it is usual to allow rays from one of unknown power and also one of known power to fall upon a prepared surface, and there to compare the illuminating effect, which, however, presents difficulties when the lights are of different tints. They propose, therefore, to interpose between the lights and the illuminated surface an arrangement which will, at will, cut off the rays of either light, but not both entirely, or else portions of each light can be obscured, leaving only visible on the illuminating service the part where the two rays come in contact. Thus a suitably slotted plate will allow both beams to fall side by side if the receiving surface is at the proper distance from the slot, while by moving the slotted plate either light can be shut off and the receiving surface only receives the ray from one. Thus with two lights, one red and one blue, the slotted plate placed at one extremity of its slide will let fall upon the receiving surface a beam of, say, red. Moving the plate slowly across the field the blue ray enters and the two colours are side by side. A further movement in the same direction and the field is entirely blue. It is found that the eye is only sensitive to these changes of colour when the plate is moved comparatively slowly; when moved rapidly to and fro only a blurred image with a throbbing effect is exhibited. This only results when the lights are of unequal intensity. When equally intense the two colours blend into a homogeneous tint and no throbbing is shown. Thus relative intensities of compared lights can be estimated either by simply comparing the two lights side by side on a receiving surface—and this only yields accurate results when the lights are nearly, or of, the same colour, and does not in any way eliminate the personal "error"—or by causing the cutting-off plate to oscillate at a suitable speed, which enables the relative intensities of lights of widely differing colours to be estimated with certainty. A finger

of wood or metal (moved in front of the receiving screen so as to cut off each light alternately) acts in the same way as the slotted plate, or a wheel with suitable vanes revolved has the same effect. In these instances the rays of light pass direct from their source to the eye, or rather to the intervening translucent screen or receiving surface.



FIG. 47.—SIGHTING WHEELS FOR SIMMANCE-ABADY'S "FLICKER" PHOTOMETER.

The above sketch shows a series of spare sighting wheels for Simmance-Abady's "Flicker" photometer. When the lights are unequal the disc (one of the wheels above) shows a rapid throbbing, but when the intensities are equalised, the disc becomes a clear, steady blend of the two colours. It was found that with lights of the same colour the arrangement affords means for a very delicate test, and until the two lights are equal in intensity the throbbing or flicker effect is very marked, which ceases on equality being obtained.

Fig. 48 shows the flicker photometer head adapted to an ordinary photometrical bar—A, sighting tube; B, box containing wheel (as shown in previous figure), divided quadrant; and it also contains a spring motor, stopping, starting, and speed regulating lever, sighting lenses or

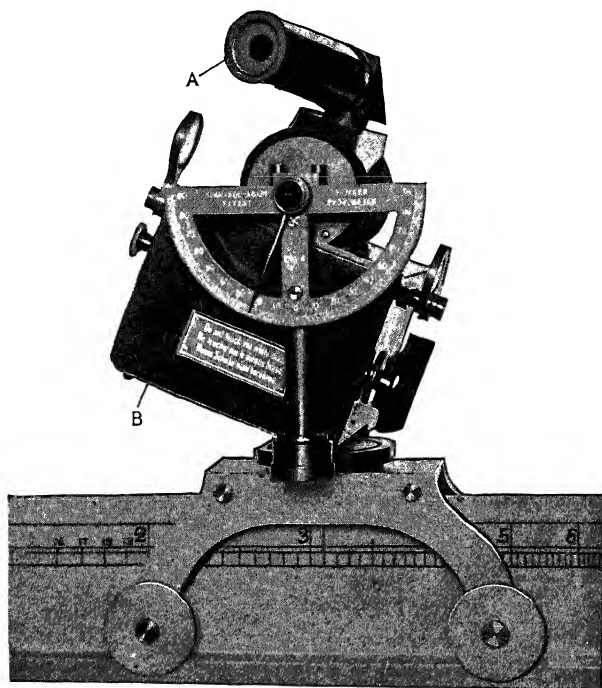


FIG. 48.—SIMMANCE-ABADY "FLICKER" PHOTOMETER HEAD.

angle finder. The method of use is simple. The white screen is allowed to revolve (the speed is adjustable). A portion of the wheel is viewed as a disc (focussed by the telescope to suit observer). When the lights are unequal, "flickering" effect is observed. When equality has been reached, the flickering ceases, and a clear, apparently



motionless disc is observed. Any difference in colour between the contrasted lights is immaterial.

**Street Photometry.**—The photometer consists of a triple chamber of mahogany containing (1) a Simmance & Abady "Flicker" head angle form, (2) a brass screen with variable opening, and (3) a pentane burner with automatic carburettor. The flicker head has already been described, and by its means the angle of light can be found. The pentane flame is fed with pentane air gas by gravity from the carburettor, and burns to a fixed height quite steadily and undisturbed by outside conditions. The brass screen divides the flame from the flicker head, and its two shutters, meeting at a line central with centre of disc, are capable of being opened and closed by means of a handle with divided drum outside the central portion of box. The adjustment can be made and read to  $\frac{1}{20}$  millimetre, and alters the light falling upon the disc by as fine an adjustment as  $\frac{1}{1000}$  of a foot candle, the maximum light being nine foot candles.

The divisions on the divided drum of handle are 1 in. per mm., running on a spiral, and can easily be read by light reflected from the pentane lamp by a mirror provided and set at a suitable angle. The photometer head itself is provided with shield from extraneous light.

To make a test with the above photometer put down view finders and shutters of flicker, adjust pentane flame to proper height (by coincidence of tip of flame with the height mark), start flicker, having wound the clock. Observe the flicker, and turn the handle with divided drum. This will vary the orifice in front of the pentane flame, and when the flicker disappears take a note of the figure on drum coincident with the arrow indicator. Make as many readings at each angle as the type of lamp necessitates; with a steady light one reading suffices, a flickering arc requires, perhaps, a dozen; in each case make a note of the figure upon the divided drum. Measure the distance

from source of light to centre of flicker disc; if, as is generally the case, this is impracticable, then note firstly the angle (not the halved angle), and either the length of perpendicular or base line, as explained. This gives the distance from the light to the disc. You thus have the following particulars :—

1. (a.) Distance of light from flicker disc in feet.
- (b.) Condition which created a balance, *i.e.*, either extent of opening of shutter, or voltage of electric lamp or both.

The remainder of the test is made in the laboratory or photometrical room afterwards, as follows :—

2. (a.) Place photometer on the saddle provided, which travels on base board provided.
- (b.) Reconstitute the conditions by opening shutter as it was at test, or regulating lamp to same voltage, or both.
- (c.) Light the Simmance 1-candle standard supplied, and place it at a suitable distance from, and in line with, centre of flicker disc.
- (d.) Turn this latter to horizontal, and move photometer (on its saddle) until equality is obtained.

Measure distance from 1-candle standard to flicker disc.

You thus obtain, in lieu of 1 (b), the distance in feet at which a candle must be held to balance the light at the particular angle tested. Q.E.F.

SIMMANCE AND ABADY'S PATENT ILLUMINATING EFFECT  
PHOTOMETER.

Table giving multipliers or factors for different angles, which factor, when multiplied by the square of the base line, gives the square of the distance from the light under test to the disc.

Angle.	Factor.	Angle.	Factor.	Angle.	Factor.	Angle.	Factor.
Degrees.		Degrees.		Degrees.		Degrees.	
10	1.026	40	1.704	52	2.638	64	5.204
15	1.070	40.5	1.729	52.5	2.698	64.5	5.395
20	1.130	41	1.755	53	2.761	65	5.599
25	1.205	41.5	1.782	53.5	2.826	65.5	5.815
30	1.333	42	1.811	54	2.894	66	6.044
30.5	1.347	42.5	1.84	54.5	2.965	66.5	6.289
31	1.36	43	1.87	55	3.039	67	6.55
31.5	1.375	43.5	1.901	55.5	3.117	67.5	6.828
32	1.39	44	1.933	56	3.198	68	7.126
32.5	1.406	44.5	1.966	56.5	3.283	68.5	7.445
33	1.422	45	2.0	57	3.371	69	7.786
33.5	1.438	45.5	2.035	57.5	3.464	69.5	8.153
34	1.455	46	2.072	58	3.561	70	8.548
34.5	1.472	46.5	2.111	58.5	3.663	70.5	8.974
35	1.49	47	2.15	59	3.77	71	9.434
35.5	1.508	47.5	2.19	59.5	3.883	71.5	9.932
36	1.527	48	2.233	60	4.0	72	10.472
36.5	1.547	48.5	2.277	60.5	4.124	72.5	11.059
37	1.568	49	2.323	61	4.254	73	11.698
37.5	1.589	49.5	2.37	61.5	4.389	73.5	12.397
38	1.610	50	2.42	62	4.537	74	13.164
38.5	1.633	50.5	2.47	62.5	4.69	74.5	14.002
39	1.656	51	2.525	63	4.849	75	14.928
39.5	1.680	51.5	2.581	63.5	5.023	...	...



The following Tables give factors for calculating spherical and hemispherical intensities, and are from a paper read by Jacques Abady entitled "Light Measurements":—

MEAN SPHERICAL INTENSITY (reading every  $10^\circ$ ).

LONGITUDES.	Readings, Candle Power.					Factor for Area.	Mean Reading × Factor.
	$0^\circ$	$90^\circ$	$180^\circ$	$270^\circ$	Mean.		
90 deg. N						0.0015	
80 "						0.014	
70 "						0.03	
60 "						0.045	
50 "						0.056	
40 "						0.069	
30 "						0.075	
20 "						0.083	
10 "						0.081	
Horizontal						0.085	
10 deg.						0.084	
20 "						0.083	
30 "						0.075	
40 "						0.069	
50 "						0.056	
60 "						0.045	
70 "						0.03	
80 "						0.014	
90 " S						0.0015	
Mean Spherical Intensity =							

MEAN SPHERICAL INTENSITY (reading every  $15^\circ$ ).

[illegible]

MEAN SPHERICAL INTENSITY (reading every  $22\frac{1}{2}^\circ$ ).

LONGITUDES.	Readings, Candle Power.					Factor for Area.	Mean Reading × Factor.
	0°	90°	180°	270°	Mean.		
90 deg. N						0.01	
67½ "						0.075	
45 "						0.14	
22½ "						0.18	
Horizontal						0.19	
22½ deg.						0.18	
45 "						0.14	
67½ "						0.075	
90 " S						0.01	
Mean Spherical Intensity =							

MEAN SPHERICAL INTENSITY (reading every 30 ).

LONGITUDES.	Readings, Candle Power.					Factor for Area.	Mean Reading × Factor.
	0°	90°	180°	270°	Mean.		
90 deg. N						0.017	
60 "						0.13	
30 "						0.223	
Horizontal						0.26	
30 "						0.223	
60 "						0.13	
90 " S						0.017	

Mean Spherical Intensity =

MEAN HEMISPHERICAL INTENSITY (reading every  $10^\circ$ ).

LONGITUDES.		Readings, Candle Power.					Factor for Area.	Mean Reading × Factor.
		0°	90°	180°	270°	Mean.		
Horizontal							0.085	
10 deg.							0.168	
20 "							0.166	
30 "							0.15	
40 "							0.138	
50 "							0.112	
60 "							0.09	
70 "							0.06	
80 "							0.028	
90 "	S						0.003	
Mean Hemispherical Intensity =								

MEAN HEMISPHERICAL INTENSITY (reading every 15°).

Readings, Candle Power.					Factor for Area.	Mean Reading × Factor.
0°	90°	180°	270°	Mean.		
Horizontal					0.14	
15 deg.					0.25	
30 "					0.224	
45 "					0.18	
60 "					0.13	
75 "					0.064	
90 " S					0.012	
Mean Hemispherical Intensity =						

MEAN HEMISPHERICAL INTENSITY (reading every  $22\frac{1}{2}^\circ$ ).

LONGITUDES.	Readings, Candle Power.					Factor for Area.	Mean Reading × Factor.
	0°	90°	180°	270°	Mean.		
Horizontal						0.19	
22½ deg.						0.36	
45 "						0.28	
67½ "						0.15	
90 " S						0.02	
Mean Hemispherical Intensity =							



The heating value of gas has been brought more to the fore of late years, owing to the large amount of gas that is used for heating purposes, such as cooking, also that now the incandescent mantle is uniformly used, and that the light obtained from these is due to the heat derived from the gas on combustion. It is important for the gas engineer to be able to ascertain the heating or calorific power of his gas. The standard of heating value of a gas is taken as follows :—

A calorie is the amount of heat required to raise 1 kilogramme (1 litre) of water 1 degree Cent.

A British thermal unit (B.Th.U.) is the amount of heat required to raise 1 lb. of water 1 degree Fahr.

Calories per cubic foot are the number of kilogrammes (litres) of water raised through 1 degree Cent. by the complete combustion of 1 cub. ft. of gas, corrected in volume to normal temperature and pressure. On the metric system it is expressed as calories per cubic metre, and is the number of kilogrammes (litres) of water raised through 1 degree Cent. by complete combustion of 1 cubic metre of gas.

British thermal units (B.Th.U.) per cubic foot are the number of lbs. of water which can be raised through 1 degree Fahr. by the complete combustion of 1 cub. ft. of gas. This formula is generally used in this country, although, owing to the convenience attained by the coincidence of kilogrammes and litres of water we use the metric water measure and the Centigrade thermometer, and calculate our results first in calories and then into B.Th.U. by multiplying by the factor 3.97.

To convert calories per cubic foot into B.Th.U. per cubic foot, multiply by 3.97 (3.968).

To convert calories per cubic foot into calories per cubic metre, multiply by 35.316.

To convert B.Th.U. per cubic foot into calories per cubic metre, multiply by 8.9.

There are three or four well-known instruments on the

market by which a calorific test can be made. These are—

1. The Simmance-Abady Patent Calorimeter for Gas.

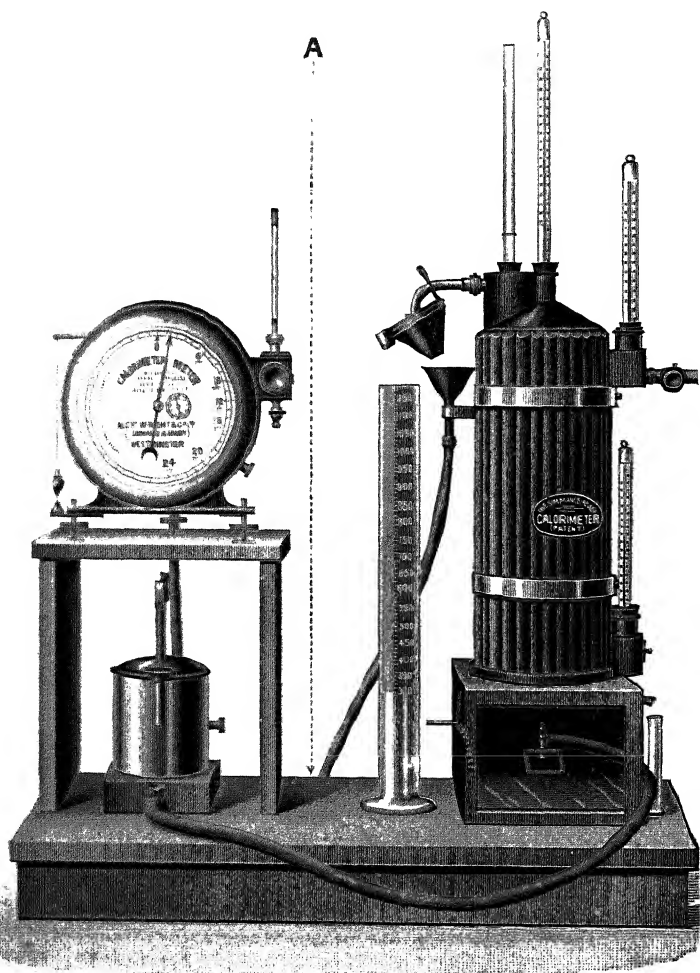


FIG. 50.—SIMMANCE-ABADY GAS CALORIMETER.

(2.) The Junker Calorimeter, invented by Herr Junker of Dressau.

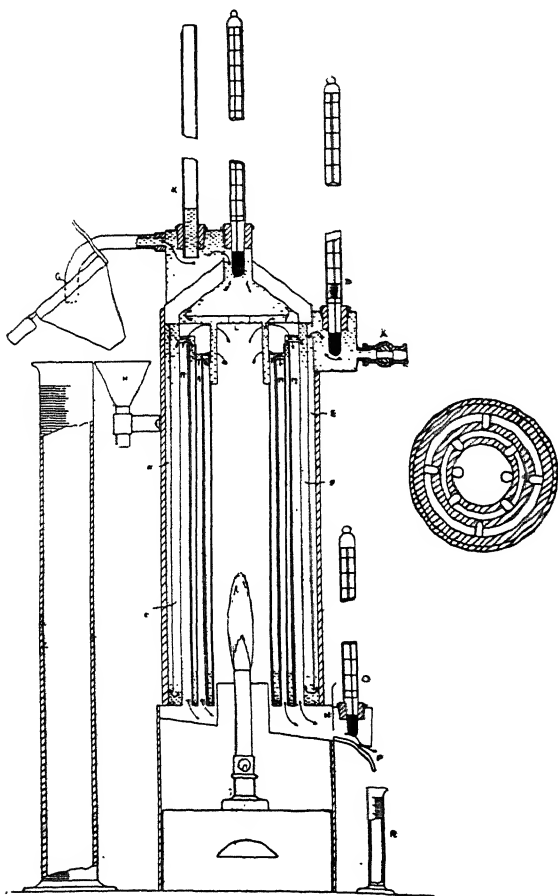


FIG. 51.—SIMMANCE-ABADY CALORIMETER—SECTIONAL ELEVATION.

(3.) The Boys Calorimeter, devised by Professor C. V. Boys, F.R.S., which is now the official instrument, and is described in the "Gas Referees' Notifications."

1. *The Simmance-Abady Patent Calorimeter for Gas.*

—The description of the apparatus is as follows, and can easily be seen by the accompanying illustrations:—

A cylindrical vertical form is adopted, and the water enters at A and passes through the fine adjustment cock B. On its entry it fills to a certain height the tube C; the height of the water is denoted by the Erdmann float. It will be found convenient to mark the height of the water in tube C. A small rubber band sprung round it is most convenient, and the water should be turned on till this level is reached, and during all tests, and each time the three thermometers are read the regularity of the water level should be verified.

After rising in the water gauge the water flows through the thermometer chamber D, thus giving the inlet temperature. From thence it fills a water jacket enveloping the whole body of the calorimeter, and which is in its turn covered with a sheet of non-conducting material and lagged closely with polished wood strips, and thus is prevented all loss by absorption or by radiation.

From the outer jacket chamber it passes down E into the calorimeter at the bottom of the cylinder, and rises up in an annular double skin (as shown by arrows), one side of which is exposed to the products of combustion at their point of exit, taking out from them the last degrees of heat left, whilst the other is the wall of the jacket chamber.

From this point the water flows into a second chamber, entering it, as in the case of the first chamber, at the bottom, being conveyed by tubes from the top of the first to the bottom of the second. Here it is in contact with the products of combustion at an earlier stage of cooling. A third chamber brings it nearer the source of heat, and at this point it envelops the flame itself. Being warmed by its previous progress, it does not cause the violent absorption of heat from the flame which would occur were it absolutely cold, and thus the structure of the flame is pre-

served, and there is no sudden condensation (to water) of products.

Finally the water mounts into the receptacle H, immediately above the flame, and receives the full first heat. In this chamber is inserted the outlet thermometer J, and from thence the water passes through K to the measuring chamber M, or when the apparatus is not in use to waste by L.

The flame is situated in the centre, well up in the calorimeter (the burner is constructed on the Bunsen principle, *i.e.*, non-luminous), in a chimney consisting of walls of water in thin copper skins. The heated products strike straight up to the top water chamber, which is filled with the hottest water, and so practically no condensation takes place at this point. Spreading out under this top chamber G, they reach the top of the outer water wall F, and there are suddenly and violently cooled, and here the greatest deposit of condensed water is made. The products find themselves over the double passage between the first and second, and second and third annular water chamber, and being unsupported and constantly increasing in weight, drop rapidly down PP, lapping over the cold surface in a clinging film. By the time they reach the bottom they are at the atmospheric temperature, and flow out through Q into measuring chamber R.

Thus we have admitted water at a given temperature, and warmed it to a certain extent by the combustion of a known quantity of gas, the heat of which gas has been entirely expended on the water (all exterior parts of the calorimeter being at the temperature of the ingoing water and absolutely no heat being lost), so that all the heat is carried off by the waterflow and registered. The products of combustion must be at the atmospheric temperature, and this is ascertained by the thermometer placed in the outlet, but not in the condensed water.

The why and wherefore of making a deduction for the amount of water condensed need not be discussed here, sufficient to say that it is done.

We will say that the combustion of  $\frac{1}{10}$  of a cub. ft. of gas has caused the condensation of 1.8 cub. cm. of water. Now, the latent heat of steam is 538 calories, *i.e.*, to evaporate 1 litre of water from boiling point to steam has absorbed as much heat as would have raised it 538 degrees Cent. without increasing its temperature. Therefore in reducing steam to 1 litre of water at 100 degrees, 538 units would have been liberated, and in bringing this down to atmospheric temperature a further 85 units should be added.

We have collected 1.8 c.c. or .0018 of a litre of condensed water, which therefore represents eighteen hundreds of 623 units = 1.1224 for  $\frac{1}{10}$  of a cub. ft., which (multiplied by 10 and deducted from the gross calories) gives us the net calories per cub. ft.

This calculation can be saved, by multiplying the number of c.c. of water condensed by .6.

*Example—*

Temperature on inlet	-	-	-	-	15.6° Cent.
„ outlet	-	-	-	-	26.8° „
„ outlet products	-	-	-	-	15.6° „
Gas passed, cub. ft.	-	-	-	-	0.10
Air temperature	-	-	-	-	15.6° Cent.
Gas temperature	-	-	-	-	15.6° „
Water collected	-	-	-	1,230 c.c. or 1.230 litres.	

$$\begin{array}{r}
 .1) 1.230 \\
 \underline{1.230} \\
 \text{Difference in temperature} = 11.2 \\
 \underline{\hspace{1cm}} \\
 2460 \\
 1230 \\
 \underline{1230}
 \end{array}$$

137.760 gross calories per cub. ft.

Water condensed = 1.8 c.c.  $\times$  0.6 = 10.8 calories to be deducted from gross = 137.76 - 10.8 = 126.96 net calories per cub. = 126.96  $\times$  3.97 = 503.63 B.Th.U (net) per cub. ft. at N.T.P.



The products of combustion pass through these tubes in a downward direction, whilst a current of water ascends outside the tubes in an opposite direction. By this arrangement all the heat given out by the combustion of the flame is absorbed by the water, and the spent gases, together with condensed water, pass out through the side conduit at the temperature of the room.

For the flat flame test, Bray's No. 7 "Economiser" over Bray's No. 4 "Regulator" as prescribed. The figures are taken from the weekly sheets, irrespective of the testing station at which they were obtained, and the average figures are the result of averaging the weekly averages of all the testing stations. The average is not a true one, but may be taken as fairly accurate.

The prescribed illuminating power for the Gas Light and Coke Co. is sixteen candles, and for the other two companies fourteen candles.

The calorific power of some of the principal gases expressed in B.Th.U. are:—

Uncarburetted water-gas from	-	-	-	-	270.296
Carburetted water-gas of 21.9 candles	-	-	-	-	624
Coal-gas 16 candles (No. 2 London Argand)	-	-	-	-	580

The calorific values of each of the combustible constituents of coal-gas, assuming that none of the steam resulting from the burning of the hydrogen is condensed to water, are:—

	Calories per cubic foot.					
Hydrogen	-	-	-	-	-	73.6
Methane	-	-	-	-	-	242.1
Ethane	-	-	-	-	-	450.1
Propane	-	-	-	-	-	645.3
Butane	-	-	-	-	-	851.1
Pentane	-	-	-	-	-	1056.3
Ethylene	-	-	-	-	-	404.3
Propylene	-	-	-	-	-	600.0
Butylene	-	-	-	-	-	821.0
Benzene (vaporised)	-	-	-	-	-	953.9
Toluene	-	-	-	-	-	1121.8
Carbon monoxide	-	-	-	-	-	85.8



With these figures and an analysis of the gas, it is possible to obtain by calculation a close approximation to the calorific value of a sample of coal-gas.

Prof. V. B. Lewes in a lecture entitled "Candles and Calories," read before the Institute of Gas Engineers in June 1903, gives the following as the constituents of coal-gas, and tabulates them according to their calorific value as follows :—

Description of Gas.	Calories per Cubic Foot.		British Thermal Units per Cubic Foot.	
	Gross.	Net.	Gross.	Net.
Benzene vapour - - -	938.9	902.5	3,718	3,574
Ethylene - - - -	404.8	381.3	1,603	1,510
Methane - - - -	258.6	232.0	1,024	919
Carbon monoxide - -	83.3	83.3	330	330
Hydrogen - - - -	82.0	68.7	325	272

Taking now an ordinary sample of 16-candle power coal-gas (old method No. 1 London Argand) as supplied to the City of London, and applying these values to its combustible constituents, we have :—

Hydrogen	-	-	-	54 per cent.	×	325 = 17,550	
Methane	-	-	-	34	„	×	1,024 = 34,816
Ethylene	-	-	-	3	„	×	1,603 = 4,809
Benzene	-	-	-	1	„	×	3,718 = 3,718
Carbon monoxide	-	-	-	6	„	×	330 = 1,980
							<hr/>
							62,873

or 628.73 B.Th.U. gross for a cubic foot; while if the calorific value be tested direct in the calorimeter, we obtain, as the value of the mean of ten determinations, by the Junker calorimeter, 157 calories per cub. ft., or  $157 \times 3,968 = 623$  B.Th.U.

Another most interesting Table given by Prof. V. B.

Lewes for the various calorific values of coal-gas and carburetted water from 12 to 20 candles, is as under :—

#### CALORIFIC VALUE OF PURE COAL-GAS.

Candle Power.	Calories.		British Thermal Units.	
	Gross.	Net.	Gross.	Net.
12	136.0	120.6	540	480
13	141.0	125.6	560	500
14	147.0	131.2	585	522
15	153.2	136.2	609	542
16	157.0	141.2	625	562
17	162.5	146.2	647	582
18	168.3	151.2	670	603
19	173.3	156.3	690	622
20	178.8	161.3	712	642

#### CALORIFIC VALUE OF CARBURETTED WATER-GAS.

Candle Power.	Calories.		British Thermal Units.	
	Gross.	Net.	Gross.	Net.
12	123.1	113.5	490	452
13	128.1	118.6	510	472
14	132.9	122.8	529	489
15	137.4	127.6	547	508
16	142.4	132.4	567	527
17	147.5	137.4	587	547
18	152.7	142.4	607	567
19	157.5	147.5	627	587
20	162.5	152.5	647	607

**Flame Temperature.**—This is a matter which has undoubtedly not received the attention (anyhow in this country) that it deserves.

Undoubtedly, the actual flame temperature, or the temperature of a flame, is not denoted by its calorific

value at present. The calorimeter takes into account the total calorific value of a gas (the net value affecting the matter considerably), whereas the temperature of a flame depends chiefly on the amount of the substances to be heated to that temperature.

In flame under consideration the only substances to be considered are nitrogen, oxygen, carbon dioxide, and water vapour. The nitrogen is the most important, being proportional to the oxygen required for combustion. The gas which requires more oxygen for combustion, and therefore produces more water of condensation, will give a lower flame temperature in relation to its calorific power.

The analysis of a gas has a great bearing on the flame temperature, for instance. Carbon monoxide has a much lower calorific power than ethylene, but gives a higher flame temperature, as more heat units are liberated in proportion to the air required for combustion, and a gas that contains a large proportion of hydrogen and a small percentage of marsh gas would give more heat by a calorimeter, but would give a lower flame temperature because of the greater specific heat of water vapour as compared with that of carbon dioxide and nitrogen.

M. Mahler, on the value of the Flame of Combustibles, says that the calorific power and the chemical composition are, in general, sufficient elements of comparison between natural combustibles. These data permit of the calculation of the value of flames; and the question may be asked of the practical utility of this. The value of the flame of combustible is the same thing as its temperature of combustion under constant pressure. It is measured by the thermometric degrees, through which the gaseous products of the combustion are raised. Supposing them to be heated by all the gases due to the combustion and solely by it, and that the combustion is complete. Now it is clear that if the calorific value, the chemical composition, and the specific heat of the gas is known, the flame temperature can be calculated.

Flame temperature is a theoretical absolute value which is not reached owing to the defective character of all heating apparatus, but nevertheless it is in many cases of the utmost value as a guide to the judgment.

Mahler gave the following formula for calculating the calorific ( $q$ ) under constant pressure of the unit weight of a fuel:—

$$(1.) q = N(C_m T_1 - C_m T_0)$$

in which  $C_m$  = the mean specific heat of one of the combustion gases between zero and  $T_0$  on the absolute scale.

$N$  = the number of molecules in "mol" volumes of 22.32 litres (the "mol" is Ostwald's term for the molecular weight in grams).

$T_0$  = the initial temperature (from the absolute zero).

$T_1$  = the final (or flame) temperature (from the absolute zero).

Specific heat increases with temperature, and the changes are so great at high temperature that they cannot be disregarded. Mallard and Le Chatelier apply the following equation to express the mean specific heat:—

$$C_m = \alpha + \beta T_1$$

in which  $\alpha$  and  $\beta$  are coefficients, having the following values for the different products of combustion:—

	$\alpha$	$\beta$
For the permanent gases (nitrogen and carbonic oxide) - - -	- 1 $6.5 \times 1,000$	- 2 $0.6 \times 1,000$
For superheated aqueous vapour	- 1 $6.5 \times 1,000$	- 2 $2.9 \times 1,000$
For carbonic acid - - -	- 1 $6.9 \times 1,000$	- 2 $3.7 \times 1,000$

Taking  $T_0$  as 273 or 0 degrees Cent., and  $\theta$  as = the final temperature on the Centigrade scale, and  $T_1$  therefore as  $= \theta + 273$ , then inserting the foregoing values in Equation (1) it becomes:—

$$(2.) q = N \left[ \frac{\alpha \theta}{1000} + \beta \frac{(\theta + 273)^2 - 273^2}{1000^2} \right].$$

The value of  $\theta$  can be found from this equation, if the number of molecules formed by the combustion and the calorific power stated in large calories are known.

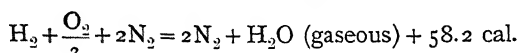
The Table shows the heat in calories of the "mol" volume of 22.32 litres under constant pressure for different gases :—

Temperature.	Nitrogen, Oxygen, Carbonic Oxide, Hydrogen.	Superheated Aqueous Vapour.	Carbonic Acid.
Degrees Centigrade.	Calories.	Calories.	Calories.
0	0.00	0.00	0.00
100	0.68	0.83	0.87
200	1.39	1.73	1.85
300	2.10	2.67	2.87
400	2.82	3.69	3.99
500	3.56	4.76	5.17
600	4.31	5.89	6.44
700	5.07	7.07	7.77
800	5.85	8.30	9.16
900	6.63	9.62	10.66
1,000	7.43	10.98	12.12
1,100	8.24	12.40	13.85
1,200	9.05	13.87	15.55
1,300	9.89	15.41	17.33
1,400	10.73	17.00	19.18
1,500	11.59	18.65	21.11
1,600	12.46	20.35	23.09
1,700	13.24	22.13	25.18
1,800	14.23	23.93	27.31
1,900	15.14	25.83	29.55
2,000	16.05	27.76	31.83
2,100	16.98	29.74	34.18
2,200	17.92	31.81	36.64
2,300	18.87	33.91	39.14
2,400	19.84	36.10	41.75
2,500	20.81	38.32	44.40
2,600	21.80	40.62	47.16
2,700	22.80	42.95	49.96
2,800	23.82	45.37	52.87
2,900	24.84	47.82	55.81
3,000	25.88	50.35	58.86

The approximate value of the flame temperature will usually be known, and trials are then made with the help of the table, taking the known number of molecules, until the temperatures in the table which give the calorific values next, above, and below the ascertained value have been found. Then for the small interval of 100 degrees between the two temperatures no practical error will be introduced, if the calorific value is taken as proportional to the temperature, and the exact value of the flame temperature thus ascertained.

The method for using the table on p. 209 (and which facilitates the rapid solution of the equation) is as follows:—

The combustion of hydrogen will serve as an example. There are four volumes of nitrogen introduced from the air with every "mol" volume of oxygen. The equation is:—



Thus the products of combustion of 1 molecule (2 grams) of hydrogen are 2 molecules of nitrogen and 1 molecule of aqueous vapour, and  $q = 58.2$  calories. Assuming the probable temperature of combustion to be between 1,800 and 2,000 degrees Cent., the calorific value is reckoned by aid of the Table for three temperatures, viz. :—

Heat.	1,800° C.	1,900° C.	2,000° C.
2 molecules of nitrogen	28.46	30.28	32.10
1 molecule of aqueous vapour	23.93	25.83	27.76
	52.39	56.11 ( $q_1$ )	59.86 ( $q_2$ )

The true calorific value of 58.2 lies between the figures marked  $q_1$  and  $q_2$ , and hence the flame temperature  $\theta$  must be between 1,900 and 2,000 degrees Cent. The difference of flame temperature over this small interval of 100 degrees may be taken as proportional to the difference of calorific

value, which is  $59.86 - 56.11 = 3.75$ , and therefore 1 calorie corresponds with a change of temperature of  $1,000 \div 3.75 = 26.7$  degrees. Consequently, the difference between the calorific value of 56.11 at 1,900 degrees and the ascertained calorific value of 58.2 corresponds with a change of temperature of  $58.2 - 56.11 = 2.09 \times 26.7 = 56$  degrees. Therefore the temperature of the hydrogen flame is  $1,900 + 56 = 1,956$  degrees Cent.

The following lists of fuels, liquids, and gases were calculated in this manner:—

Fuels.	Net Calorific Power. B.Th.U. per pound.	Flame Temperature. Degrees Cent.
<b>Solids—</b>		
Oakwood - - - -	7,860	1,865
Bohemian lignite - - -	10,060	2,020
Flaming coal from Blanzky -	14,500	1,990
Gas-coal from Saarfeld - -	14,600	1,950
Rich coal from Treuil - - -	15,450	2,010
Anthracite - - - -	14,920	2,030
Pennsylvanian anthracite - -	14,650	2,000
Peat - - - -	10,060	2,020
<b>Liquids—</b>		
Ethyl and methyl alcohol -	...	1,700
Amyl alcohol - - - -	...	1,850
American crude petroleum -	18,720	2,000
American refined petroleum -	18,500	1,660 (?)
<b>Gases—</b>		
Hydrogen - - - -	...	1,960
Carbonic oxide - - - -	...	2,100
Methane - - - -	...	1,850
Acetylene - - - -	...	2,350
Coal-gas - - - -	...	1,950
Water-gas - - - -	...	2,000

*Journal of Gas Lighting*, vol. lxxxv., p. 503.

This undoubtedly proves that the composition of the gas and the specific heat of its constituent will be of more importance in the future than at present, especially if

calorific value is going to be taken account of. One method of great assistance is to give both the gross and net calories, as the bigger the difference the lower the flame temperature.

Another interesting experiment on this matter is that carried out by Professor V. B. Lewes. He pointed out in his Cantor Lectures that he had obtained over 19 candles

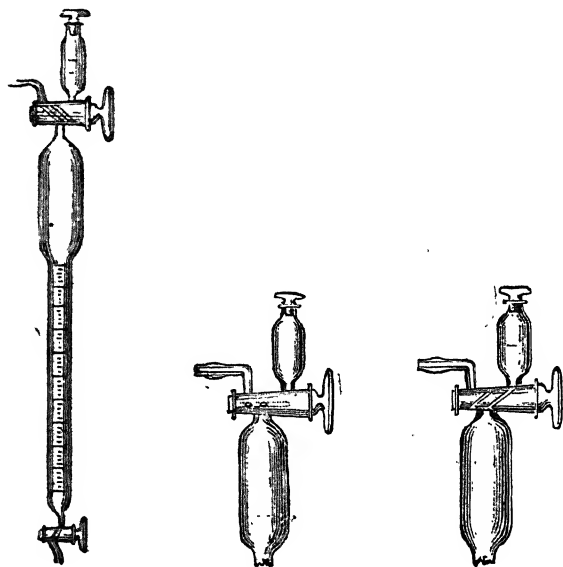


FIG. 52.—IMPROVED BUNTE BURETTE.

per cub. ft. with uncarburetted water-gas. The water-gas was purified from carbon dioxide, and had a calorific value of 81.86 calories gross and 74.66 net. This gas of 81.86 calories, or 325.7 B.Th.U., gave 19.38 candles per cub. ft. of gas consumed; the mean of four readings being 158 dles for a consumption of 8.15 cub. ft. of uncarburetted water-gas at a pressure of 1.3 in. The chimney employed 5 in. by 2 in., which gave better results than a larger



one, it being evident that under these conditions one got just the right air supply.

Coming to the question of flame temperature one finds coal-gas gives a flame the temperature of which is somewhere about 1,960 degrees Cent., and water-gas, as per above Table, is 2,000 degrees Cent.

**Gas Analysis.**—It is only our purpose to treat of the partial analysis of gas, and for the complete analysis, which is a work in itself, the reader is referred to Hempel, "Gas Analysis," pp. 44-69.

The figure opposite shows the improved form of Bunte gas burette. In the use of these burettes to arrive at any results worth considering requires a good deal of constant practice. In unskilful hands, to say the least, the results are bound to be wrong or incorrect in some way or other.

With these burettes one is capable of doing a fair analysis of a sample of gas, including :—Benzene vapours, carbon dioxide ( $\text{CO}_2$ ), heavy hydrocarbons (unsaturated, such as ethylene, &c., group  $\text{C}_n\text{H}_{m2}$ ), oxygen, carbon monoxide, hydrogen.

The marsh gas or methane can only be satisfactorily analysed in Hempel explosion pipette.

The apparatus is fitted up as above, and the following is a description and method of using it :—A and B are two burettes fitted with three-way cocks, each graduated into  $\frac{1}{5}$  c.c., and capable of holding 100 c.c.; C, a one-gallon tabulated bottle serving as water reservoir; and D, aspirator.

The burette is filled by opening the stop-cocks *g* and *k*, and allowing water to enter from the bottle C until it nearly fills the funnel *d*. The stop-cock is then closed and the indiarubber tube detached from the bottom of the burette. The longitudinal bore of the stop-cock *k* is now connected at *a* with the tube supplying the gas to be examined, and the gas aspirated into the burette by running the water out of the burette by means of the stop-

cock *g*. Rather more than 100 c.c.—about 108 c.c.—of gas should be allowed to enter the burette. This is then adjusted nearer the zero mark. By means of the bottle C sufficient water is forced into the burette through cock *g* to compress the gas, then cock *g* is closed and cock *k* is

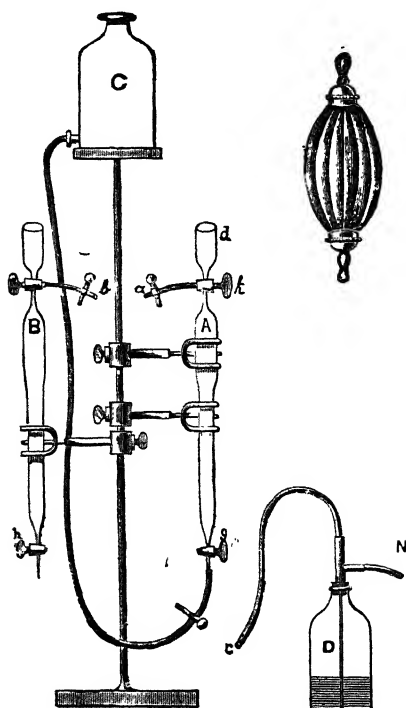


FIG. 53.—BÜNTE BURETTES AND STAND.

opened. The gas being under pressure will bubble out through the water in *d*. The funnel *d* is now filled up to the mark, stop-cock *d* is shut, and the burette is detached from stand and placed in a jar of water at the temperature of the room. The stop-cock *k* is now opened and burette left in the water for a sufficient time for the gas to become the same temperature, say ten minutes. The stop-cock *k* is now closed and the burette taken out (avoid handling as much as possible, only handling by the extreme ends) and

placed in stand. The true volume of gas in burette is now read, say 104.6 c.c.

It is not usual to estimate benzene vapour in a Bünte burette, but this can be done as follows:—The tube *c* of aspirator D is connected to the cock *g*, suction applied. Stop-cock *g* is now opened and the water in the

burette is aspirated out as low as possible without unseating the burette. The stop-cock is then closed, the tube removed, and the end of the burette dipped into a fair sized porcelain crucible containing ethyl alcohol (absolute). On opening the stop-cock *g* (the burette being under vacuo) a quantity of the solution will rise in the burette. (Care must be taken that the bottom of the burette is always immersed in the solution, for if the faintest trace of air enters the experiment is spoilt.) When the solution has entered the burette the stop-cock is closed, the burette taken from the stand, and the hand of the operator being placed firmly over *d*, the contents of the burette are shaken up with the solution. After shaking, replace burette and open stop-cock *k*. The water in *d* will now flow into burette. After allowing the water to run through burette until all the solution used has been displaced, the stop-cocks *k* and *g* are now closed, and the water level in *d* made up to the mark. The burette is now placed in the cylinder of water for ten minutes, the stop-cock *k* being opened. At the end of this period the stop-cock *k* is closed and the volume of water in burette read off. The decrease in volume of gas or increase in the water volume gives the amount of benzene vapours in the quantity of gas taken; this is easily calculated to percentages.

The next determination is for carbonic acid ( $\text{CO}_2$ ). The same procedure is gone through, but the solution used for absorbing the  $\text{CO}_2$  is strong caustic potash (1 part KHO to 2 parts  $\text{H}_2\text{O}$ ).

After the cooling of burette the difference between the volume now observed and that at the close of the last reading will give the amount of  $\text{CO}_2$  in the quantity of gas taken; this is corrected to percentage. The next absorption



FIG. 54.—COOLING JAR.

is for heavy hydrocarbons. Exactly the same procedure is gone through, but the absorbing agent is a solution of bromine. After agitating the gas with bromine great care must be taken in opening the stop-cocks. The best method is to put a porcelain basin under the burette and seal the end, and open cock *g* if the bromine vapour has caused a pressure in burette. This will prevent any loss which would occur if stop-cock *k* was opened first. Stop-cock *k* is now opened and a little potash added to absorb the bromine vapour, afterwards washing through with water, cooling, &c., as before. The difference in volume gives the unsaturated hydrocarbons in the gas.

The next absorbing is for oxygen ( $O_2$ ). The same procedure is gone through, the reagent in this case being pyrogallic acid, followed into the pipette by eight times its volume of strong KHO. This solution should not be mixed outside the burette. Many operators mix the pyro and KHO, and then add to burette; this method is not advisable. The difference in volume gives the  $O_2$  in the gas.

The next constituent to be estimated is the carbonic oxide (CO). The same procedure is again gone through, but the reagent used being a hydrochloric acid solution of cuprous chloride, the difference in volume gives the volume of CO in volume of gas taken.

When estimating for hydrogen and carbonic oxide in a sample of gas these are most conveniently estimated by combustion, as under:—

After estimating the oxygen, it is now necessary to mix the gas with an excess of air, but as the burette would not hold sufficient air to combine with the whole of the gases generally present, it is found necessary to expel a portion and work on, say, half the volume. The air is admitted by placing cock *k* in communication with the burette, opening the pinch-cock at *a*, and allowing the water to flow out at *g*. This should be continued until the water level is such that a sufficiency of air has been admitted, when the cock *g*

and the pinch-cock *a* are closed, and the contents of the burette cooled, &c., as before. The contents of the burette are now well shaken up, and after allowing to stand, the reading is taken.

Connection is then made between the burettes A and B by uniting the two at the indiarubber *a* and *b* by means of a piece of combustion tubing containing a small coil of palladium wire, the burette B having been previously filled with water. The palladium wire in the combustion tube should be brought to a red heat by means of a Bunsen burner, and the gas in the burette A is caused to pass over the heated wire into burette B by opening *g* and *h* and connecting water supply from C with the bottom of burette A. When all the gas from A has passed over (shown by the burette being full of water) the operation is reversed, the gas being again collected in A. It is then cooled as before, water level in cup *d* adjusted, and the volume read off. A solution of caustic potash is then added as in the manner described for the estimation of  $\text{CO}_2$ , and the diminution in volume noted.

Before working out the calculation it is necessary to notice what action takes place during the combustion. The gases to be dealt with (or furnace gases) are hydrogen, carbonic oxide, and nitrogen mixed with an excess of air. By passing over the red-hot palladium wire the oxygen of the air combines with carbonic oxide to form carbonic anhydride ( $\text{CO} + \text{O} = \text{CO}_2$ ), and with the hydrogen to form water ( $\text{H}_2 + \text{O} = \text{H}_2\text{O}$ ), the nitrogen of course not being affected. Now supposing that after igniting the gases and treating the residue with caustic potash a diminution in volume of 12 c.c. was observed, this would be equal to 12 c.c. of CO, for each volume of CO produces an equal volume of  $\text{CO}_2$ , and as only half the original volume taken was used, the result must be multiplied by two to give the true percentage of CO.

Supposing, also, that after ignition, but before treatment with KHO, there was a diminution in the volume of 12 c.c.,

this would be due partly to the combination of the hydrogen with the oxygen, and partly to the combination of the carbonic oxide with the oxygen.

The CO would require half its volume, or 6 c.c. of the oxygen, therefore, deducting this from the 12 c.c. due to the combustion, we have 6 c.c. as the resultant due to the combination of the hydrogen with the oxygen. Now hydrogen combines with oxygen to form water in the proportion of two volumes to one, therefore on multiplying 6 c.c. by  $\frac{2}{3}$  we have 4 c.c. as the number of c.c. of hydrogen, and this multiplied by 2 gives 8 c.c. as the percentage of hydrogen. The nitrogen is always calculated by difference.

**Specific Gravity of Gases.**—There are numerous apparatus for the determination of the specific gravity of gas. They are Bunsen Effusion Test, Letheby Specific Gravity Globe, Schilling Diffusion Test, Lux Balance, and Simmance-Abady Specific Gravity Bell.

*Bunsen Effusion Test.*—This apparatus is based on the fact that gases issuing under similar conditions of pressure from a given hole in a metallic plate flow through it at rates which vary inversely as the square roots of their densities. The time taken by a given volume of gas to pass through a small ( $\frac{1}{300}$  in. in diameter) aperture in a platinum plate, and also the time taken for the same volume of gas (such as air) of unit density to pass through under similar conditions. Then if  $T$  seconds be the time taken by the first gas, and  $T_1$  seconds the time taken by the second gas (the density of which is unity), the density of the first gas is equal to  $\frac{T^2}{T_1^2}$ . The apparatus consists of a piece of glass tube with platinum foil. There is a line scratched on this tube (which is similar to a burette turned up), and inside is a float which has two marks on it, one at the top end and the other at the bottom. The tube filled with gas and inverted into a trough containing

mercury, the tube being firmly clamped down into its position. The float is now entirely below the mercury. The stop-cock is now opened, and the gas being under pressure passes out through the orifice in the platinum foil. The surface of the mercury is carefully observed through a telescope placed level with it. As soon as the upper black line on the float appears the clock or stop-watch is started, and as soon as the second line appears the stop-watch is stopped. We now have the time taken by a fixed volume of gas to pass through the orifice. The tube is next filled with dry air and the operation repeated.

*Example—*

Time dried gas occupied in passing through orifice = 140 seconds.

$$\begin{array}{rcl}
 \text{"} & \text{"} & \text{air} & \text{"} & \text{"} & \text{"} & = 220 & \text{"} \\
 & & 220 & \times 1,400 & \times .636 & & & \\
 & & & & .636 & & & \\
 & & & & .636 & & & \\
 \hline
 & & & & .404496 & \text{sp. gr.} & & 
 \end{array}$$

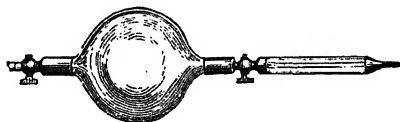


FIG. 55.—LETHEBY SPECIFIC GRAVITY GLOBE.

*Letheby Specific Gravity Globe* for determination of gas by direct weighing, requires an absolutely correct balance and considerable skill on the part of the operator.

*Schilling Diffusion Test.*—N. H. Schilling has proposed a modification of Bunsen effusion test. The tube is larger and the water takes the place of mercury.

*F. Lux Gas Balance* shows the specific gravity of gas passing through a globe by the indications of a pointer and a rider on the beam. The balance is contained in a glass case provided with adjusting screws and spirit level. The front part of the case is made to swing down, so that the whole is easily accessible. The beam of the balance swings

on agate bearings, and on either side small ivory cups filled with mercury are attached by means of brackets. These brackets or outer pillars are attached to gas pipes underneath the balance, and terminate on the left side in two stop-cocks. The handle of the balance is on the right-hand side. The beam terminates in a fine steel pointer, which moves over a quadrant scale placed parallel to the pillar by a long bracket. The beam is graduated into a

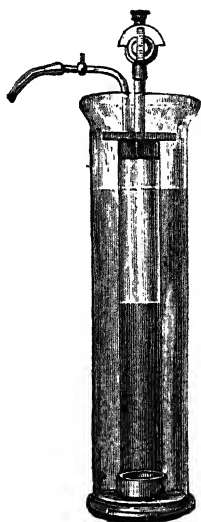
hundred divisions, with a notch at every fifth, and marked at every tenth division, beginning at the centre of the beam with the figure 0.0, 0.1, and so on up to 1.0.

The quadrant scale is divided into forty-five divisions, the one in the centre being marked 0.0, while every tenth division on either side is marked 0.1 and 0.2 respectively. Above the zero there is a plus sign (+), and below a minus sign (-).

The balance must be placed on a very firm base, and must not be exposed to sunlight or any variation in temperature, and must be exactly level.

FIG. 56.—SCHILLING'S  
SPECIFIC GRAVITY  
DIFFUSION TEST.

In performing test the mercury is first poured into ivory cups and the beam placed on its bearings, then the nickel rider is placed in the notch marked 1.0. If the balance is released, the pointer should exactly indicate zero on the quadrant scale, if not, this may be attained by means of the horizontal adjusting screw fitted to the centre of the beam. The rider is then shifted to 0.8 on the beam, and if the balance is properly sensitive each degree on the beam should correspond to one degree on the quadrant scale, the index should therefore point to plus 0.2 on the quadrant ( $0.8 + 0.2 = 1.0$ ).





In passing a gas into the apparatus the rider is placed on a figure near to the specific gravity expected, *i.e.*, in the case of coal-gas the rider would be placed at 0.5. The stop-cock should be so adjusted that nearly all the air will be expelled (under a pressure corresponding to about 25 mm. water column) after two or three minutes, and after five minutes the apparatus will therefore be filled with pure gas.

In the case of coal-gas, suppose, now, on the beam being released the index records  $+0.02$  on the quadrant,

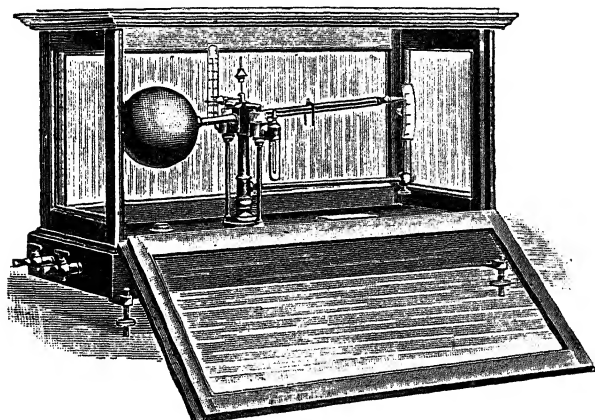


FIG. 57.—F. LUX GAS BALANCE.

the specific gravity would therefore be  $0.5 + 0.02 = 0.52$ . If, on the other hand, the index moves to  $-0.04$ , this would give the specific gravity as  $0.5 - 0.04 = 0.46$ .

The quadrant scale has twenty-two divisions on either side of the zero; one can command, with the rider in this one position (0.50), a range of specific gravity greater than any coal-gas would require.

To determine the specific gravity of gases heavier than air, the rider is set before starting on the division zero, the pointer likewise being adjusted to indicate zero, the figure 1.0 should then be added to the value found.

*Simmance-Abady Portable Specific Gravity Bell*, consisting of a small water tank in which a bell is suspended from a balance beam. The crown of the bell is drawn out

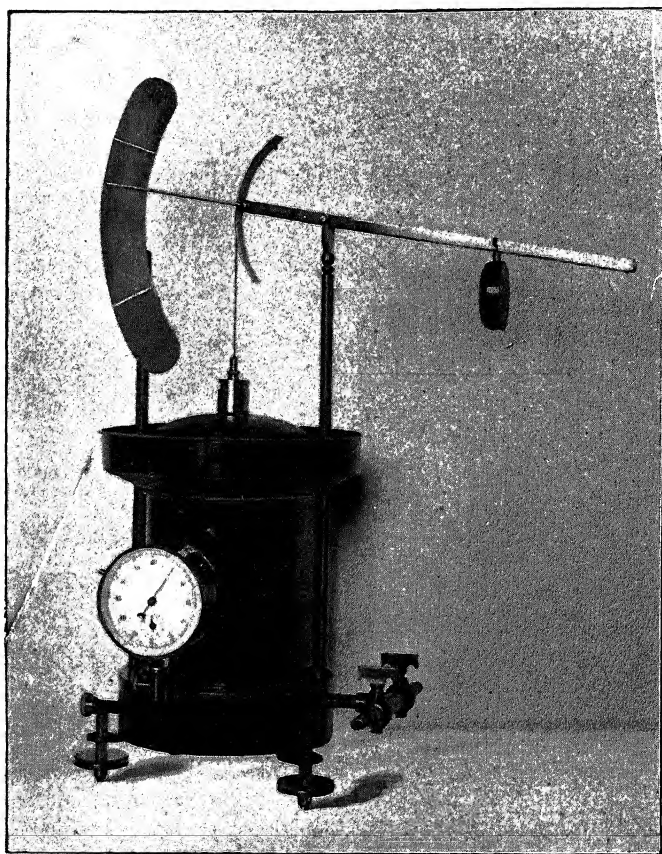


FIG. 58.—SIMMANCE-ABADY SPECIFIC GRAVITY BELL.

so that a stand pipe may be brought well up above the water line, although the bell itself is submerged up to the crown. The apex of the upper crown is formed of a small

silver plate in which a very fine effusion hole is drilled, and this is covered by a protective cap. Extended from the beam is a fine pointer, indicating upon a plate marked with two divisions. At the inlet to the stand pipe is a two-way cock for gas and air, or blow-off; the gas way to be connected by flexible or other tubing to the gas service, while the air way is free. An extra degree of exactitude is provided if the air is brought through caustic potash so as to absorb its  $\text{CO}_2$ . A reliable minute clock showing half-seconds with start, stop, and set to zero, is fixed to the tank.

*To make a Test.*—Level the apparatus by screw, seeing that the bell hangs centrally in tank. The bell by its weight is normally submerged, the tank being filled with water just to cover the dome, and only the small effusion chamber filled with air.

Set clock hand to zero, and move weight to notch at outer end of beam and slowly open air-cock, thus filling the bell with air and taking the pointer up beyond the highest mark on quadrant.

Close air-cock, hold beam with the left hand, and with the right move weight to the next notch, thus allowing the air to escape through effusion hole, and the bell to fall slowly.

As pointer passes the highest mark in the quadrant start minute clock, stopping it as the pointer passes the lowest mark, and note the time in seconds. Reset clock hand to zero, and without moving weight, slightly open gas-cock, hold down bell for a second or two (to expel the last trace of air through effusion hole), and then release same and let bell rise as before.

When pointer is well above the highest mark on quadrant turn off gas-cock. After a second or two the confined gas will have attained atmospheric pressure and the bell will fall slowly.

As pointer passes the highest mark on quadrant start minute clock, stopping it as the pointer passes the lowest

mark, and note the time in seconds. Divide the gas time squared by the air time squared.

*Example—*

Air time, 59 seconds =  $59^2 = 3,481$ .

Gas time, 41 seconds =  $41^2 = 1,681$ .

$1,681 \div 3,481 = 0.482$  specific gravity of gas.

## CHAPTER XII.

### *CARBURETTED WATER-GAS.*

THE introduction of carburetted water-gas, and the increasing headway this gas is making of late years, brings another raw material under the consideration of the gas engineer and the gas chemist's notice.

It is not intended to touch on the subject of manufacture, but only to deal with the raw materials from the chemist's point of view. The matter of coke has been already mentioned, and the remarks as to a good coke apply equally well for oil-gas manufacture as for any other purpose.

In the valuation of oils for gas-making purposes the following analyses are usually made :—

**Specific Gravity.**—Oil is generally bought by weight, and this is calculated from its volume and specific gravity. The specific gravity can be roughly estimated by specific gravity hydrometer. The specific gravity hydrometer can now be bought divided into two from 700 to 2,000 degrees. These hydrometers only give one the specific gravity roughly, and when accuracy is desired the specific gravity bottle, as shown in figure, which consists of a glass bottle holding 50 or 100 grams of distilled water at 60 degrees Fahr., and the neck is fitted with a perforated stopper, enabling the bottle to be exactly filled with the liquid with the total expulsion of all air bubbles. The oil or liquid under examination is brought exactly to the temperature of 60 degrees Fahr., the bottle is filled with the liquid, and

the stop dropped into its place, with the result that the liquid will then entirely fill the bottle and the perforation in the stopper, and any excess will flow out of the top of the stopper. The bottle is now wiped with a clean dry cloth, the top of the stopper being brushed gently with the hand so that nothing will be absorbed from the bottle, and the level of the liquid will not be lowered.

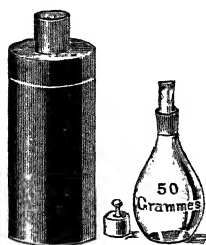


FIG. 59.—SPECIFIC GRAVITY BOTTLE.

The bottle is now weighed, and as the bottle is supplied with a counterpoise weight, the weight can be read direct. This weight divided by the weight of water contained in the bottle (viz., 50 or 100 grams, whichever may be) will give the specific gravity of the oil.

If the temperature is below 60 degrees Fahr., the bottle can be held in a vessel of water at a higher temperature, while if the temperature is above 60 degrees Fahr. (as in the warm weather) it may be reduced by means of a freezing mixture, such as adding a few crystals of ammonium chloride, or sodium thiosulphate, in water.

Sometimes it is found inconvenient to cool an oil to the exact temperature, then the specific gravity of an oil may be corrected to that temperature by means of the coefficient of expansion of the oil, which in the usual cases met with may be taken as 0.00036 per each degree Fahr.

Now the average specific gravity of oil supplied for gas-making may be taken at about 870 degrees, but supposing that the temperature was 70 degrees Fahr. instead of 60

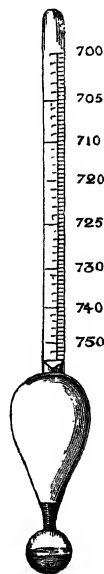


FIG. 60.—SPECIFIC GRAVITY HYDROMETER.

degrees Fahr. then the specific gravity at 60 degrees Fahr. would be found as follows:—

$$\begin{array}{r} 70 - 60 = 10 \times .00036 = .0036 \\ \hline .870 \end{array}$$

Correct specific gravity at 60 degrees Fahr. = .8736

The coefficient of expansion is an important factor when measuring the quantity of oil in stock; as the temperature of large quantities of oil stored in large tanks varies with the atmospheric temperature, it is therefore necessary to adjust for temperature by this means.

**Flash Point.** — The flash point of an oil is the temperature at which the oil commences to give off inflammable vapour. The lower the flash point of an oil, the more danger there is in the transportation, storage, and use, therefore the determination of the flash point is a matter of importance. The flash point is determined either by means of the Abel apparatus or Pensky-Marten apparatus.

The Abel apparatus is shown in illustration, and is the instrument described and used by the Board of Trade Petroleum Act of 1879. The apparatus consists of an outer jacket which is filled with water, the temperature of the water being 130 degrees Fahr. at the commencement, or, in the case of very low flash oil, cold water is used. The oil to be tested is placed in the cup provided, which is in the centre of the apparatus, and it is filled so that the top of the liquid just reaches the top of the gauge which is

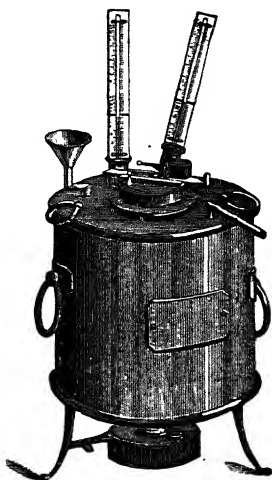


FIG. 61.—ABEL FLASH-POINT APPARATUS.

fixed within the cup. The lid of the cup with the slide closed is then put on, and the cup is placed in position. The thermometer in the lid of the cup has been adjusted so as to have its bulb just immersed in the liquid, and its position must not be altered on any account.

The test lamp is then placed (or the gas adaption is lighted) upon the lid of the cup, the pendulum or lead line is set in motion, and the rise of the thermometer in the petroleum cup is watched.

The test flame is applied once for every rise of 1 degree, the slide is slowly drawn open while the pendulum performs three oscillations, and is closed during the fourth oscillation. Directly a "flash" or light is noticed inside the cup on application of the test flame, the temperature of the thermometer in the petroleum cup gives the temperature at which the oil flashes.

It will be seen that as this instrument is water jacketed, it is not applicable for the determination of flash points of oil which have a flash point higher than 212 degrees Fahr. The apparatus used in this case is the Pensky-Marten flash-point apparatus. This ap-

paratus is shown in the illustration, and is designed for the testing of heavy oils (and it is equally suitable for testing light oils). It consists of an oil cup, with cover fitted with a Centigrade thermometer, with stirrer and air bath.

The bath is heated by a gas flame, and the slow and regular heating of the oil is ensured by the jacket of air that surrounds the cup. The cup is filled with the oil to

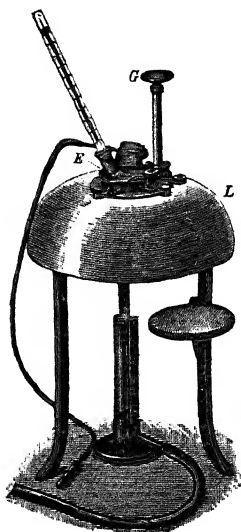


FIG. 62. — PENSKY - MARTEN  
FLASH-POINT APPARATUS.



be tested up to the ring inside, and the cover placed in position. The cup is then placed in the bath by means of the fork provided.

The thermometer is now inserted in the socket in the cover, and the gas burner lighted, the wire gauge being interposed if the temperature rises too quickly. The test jet is lighted and regulated so that the flame is only the size of a small pea.

During the heating of the oil the stirrer is used from time to time. At the increase of each degree the test flame is applied by turning the spindle G, which by a suitable mechanical arrangement opens a slide in the top of the cup, and the test flame is momentarily inserted into the cup.

Directly sufficient vapour is given off, and on inserting the test flame, a flash is noticed, the thermometer being read, which gives the flash point of the oil. The apparatus must be kept perfectly clean, as traces of moisture interfere with the flash.

The fractional distillation of an oil affords valuable information as to its suitability as an enriching agent. The apparatus employed consists of a spherical flask fitted to a Liebig's condenser. The flask must have a capacity of about twice the volume of oil to be experimented with. The procedure is as follows:—

The spherical flask is weighed empty and the weight noted. 10 oz. of the oil to be tested are now measured at 60 degrees Fahr., and poured into the flask.

The flask is now weighed, which gives the total weight of flask and oil, and deducting the weight of the flask one arrives at the weight of the oil taken.

A Centigrade thermometer which will register up to 500 degrees Cent. is fitted in the neck by means of a good well-fitting cork, through which a hole is bored to take the thermometer. The thermometer is fitted in the neck of the flask, so that the mercury bulb is on a level with the outlet of the flask.

The neck of the flask is now bound round with asbestos

string and firmly fixed in a clamp on a retort stand, and the outlet fixed on to the inlet of the Liebig's condenser as usual.

The source of heat may be supplied from a Fletcher burner, and the heat is regulated to cause the distillate to come over in separate drops at fairly regular intervals, and as the drops cease to come over, or become irregular, the heat is gradually increased.

The distillates are collected in fractions of 1 oz., or 10 per cent. of the oil experimented on. The temperature recorded by the thermometer is noted directly the contents of the flask start boiling, and is again noted directly the first drop condenses off the end of the flask, which is easily seen in the neck of the inlet of the Liebig's condenser; from thence the temperature is noted for the completion of each ounce until volatile matter ceases to come off, when the temperature is increased until nothing but coke is left in the flask. The amount of water which comes over is particularly noted. A moistened slip of lead paper is adjusted at the outlet of condenser, and replaced by a fresh piece at the end of each fraction to ascertain the varying amount of sulphuretted hydrogen given off at various temperatures, which is easily told by the depth of colour of the lead paper.

The flask on cooling is weighed, and the amount of coke calculated.

The specific gravity of each fraction is ascertained and calculated into percentage by weight on the original oil. This will enable the total weight of the distillates to be found, and these, plus the weight of the residue, should very nearly total up to the weight of the oil taken, the deficiency not being more than 1 per cent., which is set down to loss on distillation.

Some of the points which indicate the suitability of an oil for gas-making purposes are that it should be free from or only a trace at the most; that the residue left stillation does not exceed 1 per cent.; and that the

blackening of a lead paper does not occur until near the close of the distillation. The larger the proportion of the oil that comes over within a certain range of temperature, the more easily will that oil be gasified on the plant, and more permanent will be the gas formed from such oil, and it should therefore not commence to distil at too low a temperature.

*Example—*

## SAMPLE OF RUSSIAN OIL.

Specific gravity at 60° Fahr.	-	-	.8742°.
Flash point	-	-	224° Fahr.
Residue	-	-	0.12 per cent.

No. of Fraction.	Temperature, Centigrade.	Specific Gravity at 60° Fahr.	Percentage by Weight.	Colour of Distillate.	Remarks.
St. to distil.	Deg. 66	...	...	...	...
F.D.O.	146	...	...	...	...
I. -	270	.8384	9.55	very pale straw	SH <sub>2</sub> , very slight brown stain.
II. -	291	.8520	9.71	" "	" "
III. -	300	.8556	9.75	" "	SH <sub>2</sub> , a trace only.
IV. -	309	.8584	9.78	" "	" "
V. -	311	.8620	9.82	" "	" "
VI. -	330	.8650	9.85	" "	" "
VII. -	335	.8704	9.92	straw	SH <sub>2</sub> , very slight stain.
VIII. -	365	.8744	10.00	darker straw	SH <sub>2</sub> , heavier stain.
IX. -	388	.8836	10.10	" "	" "
X. -	419	.8968	10.25	reddish brown	SH <sub>2</sub> , very heavy black stain.
Residue	-	-	0.12	...	...
Loss on distillation	-	-	1.15	...	...
			100.00	...	...

Water, nil. SH<sub>2</sub>, very mild throughout distillation. Oil of good body and clear appearance, with amber colour, blue fluorescence by reflected light.

## SAMPLE OF AMERICAN OIL.

Specific gravity at 60° Fahr. - - .8633.  
Flash point - - - - 146° Fahr.  
Residue - - - - 0.62 per cent.

No. of Fraction.	Temperature, Centigrade.	Specific Gravity at 60° Fahr.	Percentage by Weight.	Colour of Distillate.	Remarks.
St. to distil.	Deg. 58	...	...	...	...
F.D.O. -	96	...	...	...	...
I. - -	284	.8092	9.37	pale straw -	SH <sub>2</sub> , very heavy, dark brown stain.
II. - -	310	.8352	9.67	" -	" "
III. - -	326	.8424	9.76	" -	" "
IV. - -	342	.8492	9.84	" -	" "
V. - -	356	.8540	9.89	straw -	" "
VI. - -	370	.8608	9.97	" -	" "
VII. - -	384	.8648	10.01	" -	" "
VIII. - -	402	.8716	10.09	darker straw -	" "
IX. - -	420	.8800	10.19	" -	" "
X. - -	425	.8832	10.23	reddish brown	" "
Residue	-	-	0.62	...	...
Loss on distillation	-	-	0.19	...	...
			100.00	...	...

Water, nil. SH<sub>2</sub>, heavy throughout distillate. Oil of a thin body and opaque appearance, slight greenish fluorescence by reflected light.

The foregoing example shows the results of a distillation test of a Russian and an American oil.

The specific gravity of the fractions is ascertained by weighing them in a very small gravity bottle, as described before.

The percentage by weight is arrived at by dividing the specific gravity of the fraction by the original specific gravity of the oil.

*Example—*

$$\begin{array}{r}
 8633).80920(9.37 \\
 \underline{77697} \\
 .32230 \\
 \underline{25899} \\
 .63310
 \end{array}$$

In a paper entitled "Composition and Valuation of Oils used for Gas Making," by Messrs Raymond Ross and Leather, read at a meeting of the Society of Public Analysts, on 14th June 1906, the authors made classical researches on the composition of petroleum from various sources, and they gave some account of these investigations carried out on various petroleums, and a description of some of the few compounds isolated from them. They ascertained the nature of some principal constituent hydrocarbons, and then proceeded to carbonise samples of pure hydrocarbons representative of these classes, with the object of ascertaining to what extent these hydrocarbons contributed to the gas-making value of an oil. The hydrocarbons specially investigated were those containing eleven carbon atoms in the molecule undecane, the eleventh hydrocarbon of the paraffin series, and is representative of the principal constituent hydrocarbons of Pennsylvanian petroleum. The corresponding hydrocarbon of the olefine series is undecylene, and this was taken as representative of the principal hydrocarbons occurring in Scotch gas oil. Decahydronaphthalene was taken as the hydrocarbon representative of the chief constituents of Texas gas oil.

Another hydrocarbon was tetrahydronaphthalene, occurring in Borneo, and to a less extent in other petroleums.

The following Table gives the constants obtained for pure hydrocarbons:—

TABLE OF CONSTANTS OBTAINED FOR PURE HYDROCARBONS.

Name.	Boiling Point. Deg.Cent.	Specific Gravity, 15/15.	N <sub>D</sub> .	Specific Refractory Power.	Valuation Figure.
Undecane - - -	194	.746	1.4182	.560	18,400
Undecylene - - -	193	.773	1.4332	.560	15,961
Decahydronaphthalene	172	.843	1.4507	.534	11,373
Tetrahydronaphthalene	205	.977	1.5712	.584	1,829
Hexahydrocymene -	161	.783	1.4323	.552	...

In Pennsylvanian oil the constituents are chiefly paraffins, but olefine and paraffinoid bodies also occur. In Caucasian oil the constituents are mainly paraffinoid hydrocarbons or naphthenes. In Texan oil the constituents consist of complex ring compounds fully hydrogenated, and Roumanian oil consists chiefly of unsaturated ring compounds.

The figures given in the next Table were obtained by gasifying the oils in a small retort, the temperature being controlled by an electrical pyrometer. The oils were cracked at different temperatures with a view of ascertaining which gave the highest value in gas. The quantity of oil gasified was about 15 c.c. in each case. The hydrocarbons were absorbed by fuming sulphuric acid. The valuation figure is obtained by multiplying the yield of gas by the percentage of hydrocarbons found in it.

## AVERAGES OF GASIFICATION RESULTS.

Name of Oil.	Temperature of Cracking.	Cubic Centi- metres of Gas at N.T.P. per Cubic Centi- metre of Oil.	Hydrocarbons absorbed by fuming H <sub>2</sub> SO <sub>4</sub> .	Valuation Figure.
	Degrees Fahr.		Per cent.	
Pennsylvanian -	{ 1,260	445.0	35.8	15,931
	{ 1,400	529.9	30.1	15,950
	{ 1,510	563.0	26.6	14,976
Kansas - -	{ 1,260	438.9	33.6	14,747
	{ 1,400	482.6	28.4	13,706
Russian - -	{ 1,260	465.7	34.2	15,927
	{ 1,510	556.0	22.8	12,677
Russian refined -	{ 1,260	429.0	31.8	13,642
	{ 1,510	518.0	28.4	14,711
	{ 1,700	550.0	21.5	11,925
Texas - - -	{ 1,130	325.0	30.1	9,783
	{ 1,260	388.3	29.8	11,571
	{ 1,400	461.8	25.3	11,684
	{ 1,450	547.7	18.6	10,187
	{ 1,510	508.8	21.1	10,736
Californian - -	{ 1,130	370.8	29.8	11,050
	{ 1,260	529.9	26.6	14,096
	{ 1,400	573.9	25.4	14,577
Roumanian - -	{ 1,130	301.3	40.3	12,083
	{ 1,260	388.8	33.3	12,947
	{ 1,400	459.7	28.6	13,148
	{ 1,450	557.0	20.2	11,251
Galician - -	1,260	452.8	35.5	16,074
Grosny - -	{ 1,130	341.8	34.6	11,826
	{ 1,260	421.9	34.6	14,598
	{ 1,400	501.6	25.8	12,941
Borneo - -	{ 1,260	301.0	26.8	8,067
	{ 1,510	472.0	17.0	8,024
	{ 1,640	495.0	15.0	7,425
Scotch - -	{ 1,130	364.8	40.3	14,701
	{ 1,260	426.4	34.2	14,583
	{ 1,450	491.5	26.0	12,780

**Bye-product.**—The bye-products of carburetted oil-gas consist of practically only one, viz., the tar, which is called "Oil-Gas Tar." The tar when first condensed on the plant contains about 50 per cent. or more of water, and in the past this quantity of water has been the cause of the trouble to dispose of or use this tar in any way. Of late years many methods have been tried to decrease this amount of water, and if it is left to stand in a tank for a sufficiently long time, the water will settle out on the top, and the tar can be drawn off from the bottom of the tank by means of a flexible hose or suction.

The tar when so treated will contain less than 5 per cent. of water, and is useful in many ways, the chief being as a fuel for boiler purposes.

Matthews and Goulden (*Gas World*, xvi., p. 625) found in water-gas tar from Russian oil :—

Benzene	-	-	-	-	-	1.19 per cent.
Toluene	-	-	-	-	-	3.83 "
Light paraffins	-	-	-	-	-	8.51 "
Solvent naphtha	-	-	-	-	-	17.96 "
Phenols	-	-	-	-	-	trace.
Middle oils	-	-	-	-	-	29.14 "
Creosote oil	-	-	-	-	-	24.26 "
Naphthalene	-	-	-	-	-	1.28 "
Anthracene (crude)	-	-	-	-	-	0.93 "
Coke	-	-	-	-	-	9.80 "
						<hr/>
						96.90 "

Although this tar appears to have a certain value which one would expect could be utilised by the tar distiller in a similar manner to coal-tar, yet they say that the oil-gas tar contains so much paraffin that the extraction of any of the other constituents is not remunerative. On distillation of a sample of oil-gas tar, the following results were obtained :—



Specific gravity at 60° Fahr.	-	-	1.0571°.
Pitch	-	-	20.48 per cent.
Water	-	-	2.60 „

No. of Fraction.	Temperature, Centigrade.	Specific Gravity of Fractions.	Percentage by Weight.	Colour of Distillate.	Remarks.
F.D.O.	Degrees. 46	...	...	...	...
I.	184	.9020	8.53	cloudy reddish	SH <sub>2</sub> , very strong.
II.	218	.9690	9.16	cloudy yellow	SH <sub>2</sub> , " " "
III.	250	.9840	9.31	clearer yellow	SH <sub>2</sub> , " " " on cooling turned nearly solid, due to naphthalene.
IV.	280	1.004	9.50	clear yellow	SH <sub>2</sub> , very strong.
V.	312	1.020	9.64	" "	" "
VI.	350	1.046	9.89	clear reddish	" "
VII.	368	1.050	9.93	" "	" "
VIII.	376	1.067	10.01	clear red	" "
Water	-	-	2.60	...	...
Residue	-	-	20.48	...	...
Loss on distillation	-	-	0.95	...	...
			100.00	...	...

This example shows about the average, but the quality of tar differs considerably according to the oil used, temperature of carburetter, &c. The use of oil-gas tar as a fuel for boilers is well known, it gives a good result, and when properly burnt, &c., gives no smoke, which is a great inducement in some quarters for its adoption.

In *The Surveyor and Municipal County Engineer* for the 29th June 1906, appears a report on the application of oil-gas tar for improving road surfaces. This material was found very suitable for the preservation of macadam and other roadway and footway surfaces, and for preventing the formation of dust, and the penetration of wet into the crust of the road.

The trials were carried out by the Borough Engineer at Tunbridge Wells. The roads were first of all swept clean

of all loose dust, &c., and it was found that traffic could pass over the road directly afterward, and that therefore there was no closing of the road. Oil-gas tar, as proved by these experiments, appears to be probably the most effective, economical, and most expeditiously applied dust preventive treatment yet utilised in a practical way over large areas.

## APPENDIX A.

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### METROPOLIS GAS.

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#### NOTIFICATION OF THE GAS REFEREES FOR THE YEAR 1907.

OFFICE OF THE METROPOLITAN GAS REFEREES,  
66 VICTORIA STREET, S.W.,  
*December 1906.*

WHEREAS the undersigned have been appointed "Gas Referees" under the City of London Gas Act, 1868; The South Metropolitan Gas Light and Coke Company's Act, 1869; The Commercial Gas Act, 1875; The Gas Light and Coke Company Act, 1876; The South Metropolitan Gas Light and Coke Company's Act, 1876; The Gas Light and Coke and other Gas Companies Acts Amendment Act, 1880; and the London Gas Act, 1905.

And whereas it is the duty of the said Gas Referees, under the same and other Acts of Parliament, among other things, to prescribe and certify the situation and number of the testing places, and the apparatus and materials therein, for testing the illuminating power, calorific power, purity, and pressure of the gas to be provided by the Companies; and the mode to be adopted for testing and recording the illuminating power, calorific power, purity, and pressure of the gas; and the number of the times of testing, except in the case of testings for pressure and of testings made under section 5 of the London Gas Act, 1905:

Now, therefore, in compliance with the provisions of the said Acts, the said Gas Referees do hereby prescribe and certify as follows; that is to say,

As to the

#### TESTING PLACES.

The testing places shall, for the present, be as follows:—

*For the Gas Light and Coke Company.*

1. At No. 106 Fenchurch Street, E.C.
2. At No. 93 Aldersgate Street, E.C.
3. At No. 7 Tudor Street, Blackfriars, E.C.
4. At No. 123 Ladbroke Grove, Notting Hill, W.
5. At No. 3 Vincent Terrace, Islington, N.
6. At No. 1 Carlyle Square, Chelsea, S.W.
7. At No. 170 Camden Street, Camden Town, N.W.
8. At No. 14a Graham Road, Dalston, N.E.
9. At No. 47 Kingsland Road, N.E.
10. At the Offices of the London County Council, Spring Gardens, S.W.
11. At No. 1 Vinery Villas, North Bank, Regent's Park, N.W.
12. At No. 116 Lambeth Road, S.E.
13. At No. 121 Hornsey Road, Holloway, N.
14. At No. 66 George Street, Hampstead Road, N.W.

*For the Commercial Gas Company.*

1. At No. 6 Wellclose Square, St George's, E.
2. At No. 24 Parnell Road, Old Ford, E.

*For the South Metropolitan Gas Company.*

1. At No. 104 Hill Street, Peckham, S.E.
2. At No. 37 Bedford Road, Clapham Road, S.W.
3. At No. 1 Stoney Lane, Tooley Street, S.E.
4. At No. 180 Lewisham Road, Lewisham, S.E.
5. At No. 107 Blackfriars Road, S.E.
6. At No. 211 Burrage Road, Plumstead, S.E.

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As to the

## SERVICE PIPES TO THE TESTING PLACES.

The conditions to be observed in connecting the Gas Companies' mains with the apparatus in the testing places and in providing for shutting off the gas in case of emergency are prescribed by section 8 of the London Gas Act, 1905.

If obstruction of the service pipe is found, or if there is reason to think that the quality of the gas is suffering from any change occurring within the service pipe, the service pipe may be washed

out in the presence of and by arrangement with the Gas Examiner, either with hot water alone or with any usual solvent such as benzol, naphtha, or petroleum, but the use of such solvents is to be followed by a washing with hot water. In every case where the service pipe is washed out the Gas Company shall send a letter to the Gas Referees explaining why the washing was considered necessary. The Gas Companies may, if they think fit, provide a tap and funnel in any testing place for the purpose of such washing out.

No testing for illuminating power is to be made until after the lapse of an hour since the last washing out.

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As to the

STANDARD LAMP TO BE USED FOR TESTING ILLUMINATING  
POWER.

The standard to be used in testing the illuminating power of gas shall be a Pentane 10-candle Lamp which has been examined and certified by the Gas Referees. A description of the lamp is given in Appendix A. The residue of pentane in the saturator shall, at least once in each calendar month, be removed, and shall not be used again in any testings.

The pentane to be used in this lamp shall be prepared as described in Appendix B, and shall show when tested the properties there specified.

All pentane provided by the Gas Companies will be examined and certified by the Gas Referees, and will be sent to the testing places in cans, which have been both sealed and labelled by them; and no pentane shall be used in the testing places other than that which has been thus certified.

The procedure to be followed in the issue of pentane to the testing places is described in Appendix C.

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As to the

TIMES AND MODE OF TESTING FOR ILLUMINATING POWER.

1. *Testings with the Metropolitan Argand Burner, No. 2.*

The testings for Illuminating Power made with the Standard Argand shall be three in number daily. "The tests for illuminating power shall be taken at intervals of not less than one hour." "The average of all the testings at any testing place on each

day of the illuminating power of the gas supplied by the Company at such testing place shall be deemed to represent the illuminating power of such gas on that day at such testing place." (Gas-light and Coke and other Gas Companies Acts Amendment Act, 1880, sections 7 and 8.)

But "If on any one day the gas supplied by the Company at any testing place is of less illuminating power to an extent not exceeding one candle than it ought to be, the average of all the testings made at such testing place on that day and on the preceding day and on the following day shall be deemed to represent the illuminating power of the gas on such one day at such testing place." (London Gas Act, 1905, section 4 (3).)

The gas supplied by the Gas Light and Coke Company is required to have an illuminating power of 16 candles, and the gas supplied by the South Metropolitan Gas Company and by the Commercial Gas Company an illuminating power of 14 candles. (London Gas Act, 1905, section 4 (1), (2).)

The Photometer to be used in the testing places shall be the Table Photometer described in Appendix D. The air-gas in the lamp is to be kept burning so that the flame is near its proper height for at least ten minutes before any testing is made. At the completion of every testing the air-gas is to be turned off; but if the interval between two testings does not much exceed one hour and the Gas Examiner is present during the interval, he may, instead of turning it off completely, turn it down low.

The Argand Burner attached to each Photometer shall be a standard burner called the Metropolitan Argand Burner, No. 2, which has been devised by Mr Charles Carpenter. A description of the burner is given in Appendix E. No Argand Burner shall be used for testing the illuminating power of gas that does not bear the lead seal of the Gas Referees.

A clean chimney is to be placed on the burner before each testing, and care should be taken that the glass does not become dimmed by the smoking of the flame.

The gas under examination is to be kept burning, at about the usual rate, for at least fifteen minutes before any testing is made; the damper shall not be in action during this interval. No gas shall pass through the meter attached to the Photometer except that which is consumed in testing or during the intervals between the testings made on any day, and that which is used in proving the meter. (See p. 250 and Appendix N.)

The paper used in the photoped of the Photometer shall be white in colour, unglazed, of fine grain and free from water marks. It shall be as translucent as is possible consistently with its being sufficiently opaque to prevent any change in the apparent relative

brightness of the two portions of the illuminated surface when the head is moved to either side. This paper should, when not in use, be covered to protect it from dust; and if it has been in any way marked or soiled a fresh piece is to be substituted.

Each testing shall be made as follows:—

The index of the regulating tap shall be so adjusted that the meter hand makes one complete revolution in not less than 59 or more than 61 seconds. The damper for regulating the air-supply to the burner shall be screwed upwards until the flame is on the point of tailing above the chimney and then immediately be turned down only so far as to ensure that the flame burns without any smoking. The connecting rod shall now be pushed to and fro by the Gas Examiner until the illumination of the photoped by the two sources of light is judged to be equal. A balance is best attained by making small alternations of decreasing amplitude rather than by a very slow movement in one direction only. The reading on the photometric scale shall be noted. This observation is to be made four times in all, and the mean of the results taken. The time that the meter hand takes to make exactly two revolutions shall then be observed by the aid of a stop-clock or stop-watch. The mean of the four readings of the photometric scale shall be multiplied by the number of seconds in the time recorded and by the Aerorthometer reading and divided by 120. The quotient is the illuminating power.

If the gas is so rich that it cannot be made to burn at the prescribed rate without tailing above the chimney or smoking, or if the burner cannot be pushed far enough away to produce equality of illumination on the photoped, the rate must be reduced until the flame burns properly within the chimney or a balance is produced when the burner is at the far end of the slide. In all other respects the testing and calculation shall be made as described.

If, in very exceptional circumstances, the Aerorthometer scale or the table does not include the conditions that are met with, the Gas Examiner shall, in calculating the illuminating power, use the formula printed below the table.

Each testing place must be provided with a standard clock that will go for a week without re-winding.

The Gas Examiner shall, at least once a week, compare the stop-clock in the testing place with the standard clock or with his watch.

The Gas Examiner shall enter in his book the particulars of every testing of illuminating power made by him at the testing places, during or immediately after such testing; and in the case of any testing which he rejects he shall also state the cause of

rejection. No testing is to be rejected on the ground that the result seems improbable.

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*2. Testings with the Standard Flat Flame Burner.*

The testings for illuminating power made with the flat flame burner shall be made at such times as the Controlling Authority shall direct. The burner shall be Bray's "No. 7 Economiser" fitted over a Bray's "No. 4 Regulator," as described in Appendix D. The testings made with it shall be conducted in the same way as those with the Argand; but when a testing with the Argand has been made immediately before, the testing with the flat flame burner may be made when the gas has been burning through it at the usual rate for five minutes. A new burner shall be used every week.

If the gas is so poor that the burner cannot be brought near enough to produce equality of illumination on the photoped, the rate of consumption must be increased, until a balance is produced when the burner is at the near end of the slide. In all other respects the testing shall be carried out as described.

---

As to the

TIMES AND MODE OF TESTING FOR SULPHURETTED  
HYDROGEN.

"The gas supplied by the Company shall not exhibit any trace of sulphuretted hydrogen when tested in a mode to be from time to time prescribed and certified by the Gas Referees for testing and recording the presence of sulphuretted hydrogen, which mode shall not be more stringent than the mode prescribed in Schedule A of the Gasworks Clauses Act, 1871." (London Gas Act, 1905, section 6.)

The apparatus to be used in testing gas for the presence of sulphuretted hydrogen is figured in Appendix H. The gas as it leaves the service pipe shall be passed through the glass vessel in which are suspended slips of bibulous paper which have been recently moistened by dipping them in a solution consisting of 6.5 grams (100 grains) of crystallised acetate of lead dissolved in 100 cubic centimetres of water.

One testing shall be made daily.

In making the testing, gas shall be turned on to the apparatus, 1 lit at the burner as soon as the air has been swept out. When the gas has burnt for three minutes it is to be turned off, and one of the slips of paper is to be compared with another similar



slip which has not been exposed to the gas. The gas is to be taken as exhibiting a trace of sulphuretted hydrogen if the slip of paper which has been exposed to it is unmistakably the darker of the two.

In this event two of the test-slips which have been exposed to the gas shall be placed in a stoppered bottle and kept in the dark at the testing place; one of the remaining slips shall be forwarded with each daily Report (Appendix O), and the comparison slip shall be retained by the Gas Examiner for the use of the Chief Gas Examiner.

The Gas Examiner in making his return shall write either "present" or "absent" as the case may be.

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As to the

MODE OF TESTING FOR SULPHUR COMPOUNDS OTHER THAN  
SULPHURETTED HYDROGEN.

This testing shall be made on such days as the Controlling Authority shall direct. A description of the apparatus to be employed is given in Appendix K. It is to be set up in a room or closet where no other gas is burning. The gas shall pass through a meter by reference to which the rate of flow can be adjusted, and which is provided with a self-acting movement for shutting off the gas when ten cubic feet have passed.

Pieces of sesqui-carbonate of ammonia, from the surface of which any efflorescence has been removed, are to be placed round the stem of the burner. The index of the meter is to be then turned forward to the point at which the catch falls and will again support the lever-tap in the horizontal position. The lever is made to rest against the catch so as to turn on the gas. The index is turned back to a little short of zero, and the burner lighted. When the index is close to zero the trumpet-tube is placed in position on the stand and its narrow end connected with the tubulure of the condenser. At the same time the long chimney-tube is attached to the top of the condenser.

As soon as the testing has been started, a first reading of the Aerorthometer is to be made and recorded, and a second reading as near as may be to the time at which the gas is shut off. The rate of burning, which with practice can be judged very nearly by the height of the flame, is to be adjusted, by timing the index of the meter, to about half a cubic foot of gas per hour.

After each testing, the flask or beaker, which has received the liquid products of the combustion of the ten cubic feet of gas, is to be emptied into a measuring cylinder and then replaced to

receive the washings of the condenser. Next the trumpet-tube is to be removed and well washed out into the measuring cylinder. The condenser is then to be flushed twice or thrice by pouring quickly into the mouth of it 40 or 50 cubic centimetres of distilled water. These washings are brought into the measuring cylinder, whose contents are to be well mixed and divided into two equal parts.

One-half of the liquid so obtained is to be set aside, in case it should be desirable to repeat the determination of the amount of sulphur which the liquid contains.

The other half of the liquid is to be brought into a flask, or beaker covered with a large watch-glass, treated with Hydrochloric Acid sufficient in quantity to leave an excess of acid in the solution, and then raised to the boiling point. An excess of a solution of Barium Chloride is now to be added, and the boiling continued for five minutes. The vessel and its contents are to be allowed to stand till the Barium Sulphate has settled at the bottom of the vessel, after which the clear liquid is to be as far as possible poured off through a paper filter. The remaining liquid and Barium Sulphate are then to be brought on to the filter, and the latter is to be well washed with hot distilled water. (In order to ascertain whether every trace of Barium Chloride and Ammonium Chloride has been removed, a small quantity of the washings from the filter should be placed in a test tube, and a drop of a solution of Silver Nitrate added; should the liquid, instead of remaining perfectly clear, become cloudy, the washing must be continued until on repeating the test no cloudiness is produced.) Dry the filter with its contents, and transfer it into a weighed platinum crucible. Heat the crucible over a lamp, increasing the temperature gradually, from the point at which the paper begins to char, up to bright redness.\* When no black particles remain, allow the crucible to cool; place it when nearly cold in a desiccator over strong Sulphuric Acid, and again weigh it. The difference between the first and second weighings of the crucible will give the number of grains of Barium Sulphate. Multiply this number by 11 and divide by 4; the result is the number of grains of sulphur in 100 cubic feet of the gas.

This number is to be corrected for the variations of temperature and atmospheric pressure in the manner indicated under the head of Illuminating Power, with this difference, that the mean of the first and second Aerorthometer readings shall be taken as the reading.

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\* An equally good and more expeditious method is to drop the filter with its contents, drained but not dried, into the red-hot crucible.

The correction by means of the Aerorthometer reading may be made most simply and with sufficient accuracy in the following manner:—

When the Aerorthometer reading is between .955-.965, .965-.975, .975-.985, .985-.995, diminish the number of grains of Sulphur by 4, 3, 2, and 1 per cent.

When the Aerorthometer reading is between .995-1.005, no correction need be made.

When the Aerorthometer reading is between 1.005-1.015, 1.015-1.025, 1.025-1.035, increase the number of grains of Sulphur by 1, 2, and 3 per cent.

Example:—

Grains of Barium Sulphate from 5	
cubic ft. of Gas	10.4
Multiply by 11 and divide by 4	11
	<hr/>
	4)114.4

Aerorthometer reading, 1.018
---------------------------------

Grains of Sulphur in 100 cubic ft. of	
Gas (uncorrected)	28.60
Add $28.6 \times \frac{1}{100} =$	.57
	<hr/>

Grains of Sulphur in 100 cubic ft. of	
Gas (corrected)	29.17

Result :  
29.2 grains.

The Aerorthometer reading is the reciprocal of the Tabular Number. The Gas Examiner shall, not less often than once a month, compare the Aerorthometer reading with the reciprocal of the Tabular Number deduced from observations of the Barometer and Thermometer, and if there is a difference of more than one-half per cent. the Aerorthometer is to be readjusted.

As to the

#### MODE OF TESTING THE CALORIFIC POWER OF THE GAS.

This testing shall be made on such days as the Controlling Authority shall direct.

The Calorimeter to be used in testing the calorific power of the gas shall be one which has been examined and certified by the Gas Referees. A description of the Calorimeter is given in Appendix L.

In order to test the gas for calorific power, the gas shall first pass through a meter and a balance governor of the same construction as those on the photometer table. It shall then be led to the gas inlet in the base of the Calorimeter. The gas shall be

turned on and lighted, and the tap of the Calorimeter shall be so adjusted as to allow the meter hand to make one turn in from 60 to 75 seconds. The water shall be turned on so that when the regular flow through the Calorimeter has been established a little may pass the overflow of the funnel and trickle over into the sink. Water must be poured in through one of the holes in the lid until it begins to run out at the condensation outlet. The Calorimeter may then be placed upon its base. The measuring vessel carrying the change-over funnel shown in Figs. 16 and 18, pp. 277 and 278, should then be placed in position in the sink so that the outlet water is led into the sink. The hot-water outlet tube of the Calorimeter should be above but should not touch the change-over funnel. After an interval of not less than twenty minutes the Gas Examiner, after bringing the reading glasses into position on the thermometers used for measuring the temperature of the inlet and outlet water, shall then make the following observations. When the meter hand is at 75 he shall read the inlet temperature; when it reaches 100 he shall move the funnel so as to direct the outflow into the measuring vessel and at the same time he shall start the stop-clock or a stop-watch. When the meter hand reaches 25 he shall make the first reading of the outlet temperature. He shall continue to read the outlet temperature at every quarter turn until fifteen readings have been taken. The meter hand will then be at 75. He shall also at every turn of the meter except the last make a reading of the inlet temperature when the meter hand is between 75 and 100. When the meter hand reaches 100 after the last outlet temperature has been read, the Gas Examiner shall shift the funnel so as to direct the outlet water into the sink again and at the same time stop the clock or watch. The barometer and the thermometers showing the temperatures of the effluent gas, of the air near the Calorimeter and of the gas in the meter, shall then be read. The time shown by the stop-clock shall be recorded. The mean of the four readings of the inlet temperature is to be subtracted from the mean of the fifteen readings of the outlet temperature and the difference is to be multiplied by 3 and by the number of litres of water collected and the product is to be divided by the tabular number. The difference in degrees Centigrade of the temperature of the effluent gas and of the surrounding air shall be taken, and one-sixth of this difference shall be added to the result previously found if the effluent gas is the warmer of the two, or subtracted if the effluent gas is the cooler of the two.\* The result is the gross calorific power of the gas in Calories per cubic foot.

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\* This correction has been found by experiment.

In addition to the observations described, the amount of condensed water resulting from the combustion of the gas shall be measured. For this purpose the condensation water shall be led into a flask not less than twenty minutes after the Calorimeter has been placed in position. The amount collected in not less than thirty minutes shall be measured, the time of collection having been accurately noted.

The number of cubic centimetres collected shall be multiplied by the number of seconds in the time indicated by the stop-clock and by the number 1.86. The number of seconds in the time during which the condensed water was being collected shall be multiplied by the tabular number. The first product shall be divided by the second. The quotient is to be subtracted from the gross calorific power. The difference is the net calorific power in Calories per cubic foot. The gross and net calorific power in British Thermal Units can be obtained by multiplying the corresponding numbers of Calories by 3.968.

A form on which the Gas Examiner may conveniently set down his observations and the whole of the figures needed for the calculation is given at end of Appendix L. The figures in italic type are specimen figures, and represent such as might be written by the Gas Examiner.

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As to the

MODE OF TESTING THE PRESSURE AT WHICH  
GAS IS SUPPLIED.

Testings of pressure shall be made at such times and in such places as the Controlling Authority may from time to time appoint (Gas Light and Coke and other Gas Companies Acts Amendment Act, 1880, Section 6). In order to make this testing the Gas Examiner shall unscrew the governor and burner of one of the ordinary public lamps, and shall attach in their stead a portable pressure gauge. In places where incandescent burners are used for street lighting, one street lamp in each street or group of streets may be provided under the lantern with a branch closed by a screw stopper. The Gas Examiner shall in such cases connect the pressure gauge by screwing to it an L-shaped pipe fitted with a union, by means of which it may be connected to the service pipe in the place of the screw stopper. The L-shaped pipe is to be of such dimensions as to enable the pressure gauge to be fixed outside the lantern but at about the same level as the incandescent burner. It should be provided with a tap.

The gauge to be used for this purpose consists of an ordinary

pressure gauge enclosed in a lantern, which also holds a candle for throwing light upon the tubes and scale. The difference of level of the water in the two limbs of the gauge is read by means of a sliding scale, the zero of which is made to coincide with the top of the lower column of liquid (see Appendix M).

The Gas Examiner having fixed the gauge gas-tight, and as nearly as possible vertical on the pipe of the lamp, and having opened the cocks of the lamp and gauge, shall read and at once record the pressure shown. From the observed pressure one-tenth of an inch is to be deducted to correct for the difference between the pressure of gas at the top of the lamp column and that at which it is supplied to the basement of neighbouring houses.

The pressure prescribed in the Acts of the three Metropolitan Gas Companies is to be such as to balance from midnight to sunset a column of water not less than six-tenths of an inch in height, and to balance from sunset to midnight a column of water not less than one inch in height.

#### METERS.

The meters used for measuring the gas consumed in making the various testings shall be wet meters constructed with measuring drums which allow one-twelfth of a cubic foot of gas to pass for every revolution. A hand is fastened directly to the axle of the drum and passes over a dial divided into one hundred equal divisions. The dial and hand are protected by a glass. In the meter employed in testing the purity of gas the pattern of dial for showing the number of revolutions and the automatic cut-off hitherto in use shall be retained, but in the meters employed for testing illuminating power and calorific power, only the dial above described is needed. The meters should be provided with Fahrenheit thermometers. The stop-clock may be either attached to the meter or separate.

The meters used for measuring the gas consumed in making the various testings shall have been certified by the Referees, and shall, at least once in seven days, be proved by the Gas Examiners by means of the Referees' one-twelfth of a cubic foot measure. A description of this Instrument, with directions how to use it, is given in Appendix N.

The results of the testings for illuminating power, calorific power, purity, and pressure shall be recorded in the form given in Appendix O, and delivered as provided in Section 11 of the Gas Light and other Gas Companies Acts Amendment Act, 1880, and Section 5 (2) of the London Gas Act, 1905.

These regulations shall be in force from the 1st January 1907 until they are superseded by a subsequent Notification.

C. V. BOYS,	}	<i>Gas Referees.</i>
J. S. HALDANE,		
A. VERNON HARCOURT,		

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## APPENDIX A.

### THE TEN-CANDLE PENTANE LAMP.

Mr Harcourt's Ten-Candle Pentane Lamp is one in which air is saturated with pentane vapour, the air-gas so formed descending by its gravity to a steatite ring burner. The flame is drawn into a definite form, and the top of it is hidden from view, by a long brass chimney above the steatite burner. The chimney is surrounded by a larger brass tube, in which the air is warmed by the chimney, and so tends to rise. This makes a current which, descending through another tube, supplies air to the centre of the steatite ring. No glass chimney is required, and no exterior means have to be employed to drive the pentane vapour through the burner.

Figure 1 shows the general appearance of the lamp. The saturator A is at starting about two-thirds filled with pentane.\* It should be replenished from time to time, so that the height of liquid as seen against the windows may not be less than one-eighth of an inch. The saturator A is connected with the burner B by means of a piece of wide indiarubber tube. The rate of flow of the gas can be regulated by the stop-cock  $s_2$ , or by checking the ingress of air at  $s_1$ . For this latter purpose a metal cone, acting as a damper, is suspended by its apex from one end of a lever, to the other end of which is attached a thread for moving the cone up or down. The lever is supported by an upright arm clamped to the upper end of the stop-cock immediately beneath the cone. From the top of the lamp the thread descends to a small pulley on the table, and thence passes horizontally to the end of a screw moving in a small block, by turning which the gas examiner can regulate the lamp without leaving his seat. It is best so to turn the stop-cock  $s_2$  as to allow the flame to be definitely too high, but not to turn it full on, before letting down

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\* *Caution.*—Pentane is extremely inflammable; it gives off at ordinary temperatures a heavy vapour which is liable to ignite at a flame at a lower level than the liquid. *The Saturator must never have pentane poured into it when in position, if the lamp or the gas of the photometer is alight.*

the regulating cone to its working position. Both stop-cocks should be turned off when the lamp is not alight.

The chimney tube *c c* should be turned so that no light

passing through the mica window near its base can fall upon the photoped. The lower end of this tube should, when the lamp is cold, be set 47 millimetres above the steatite ring burner. A cylindrical boxwood gauge, 47 millimetres in length and 32 in diameter, is provided with the lamp to facilitate this adjustment. The exterior tube *D* communicates with the interior of the ring burner by means of the connecting box above the tube *E* and the bracket *F* on which the burner *B* is supported. A conical shade *G* is provided. This should be placed so that the whole surface of the flame beneath the tube *c* may be seen at the photoped through the opening.

The lamp should be adjusted by its levelling screws so that the tube *E*, as

tested with a plumb-line, is vertical, and so that the upper surface of the steatite burner is 353 millimetres from the table. A gauge is provided to facilitate this latter measurement. The tube *c* is brought centrally over the burner by means of the

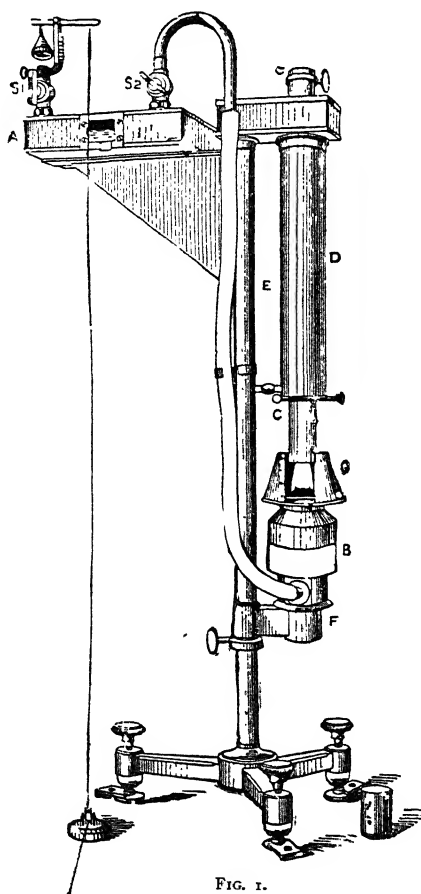


FIG. 1.



three adjusting screws at the base of the tube D. These three screws should not be quite screwed up, but only sufficiently so to keep the chimney tube central. The adjustment is facilitated by means of the boxwood gauge.

When the lamp is in use the stop-cocks are to be regulated so that the tip of the flame is about half-way between the bottom of the mica window and the crossbar. A variation of a quarter of an inch either way has no material influence upon the light of the flame. The saturator A should be placed upon the bracket as far from the central column as the stop at the end will allow. If it is found that, after the lamp has been lighted for a quarter of an hour, the tendency of the flame is to become lower, the saturator may be placed a little nearer the central column.

To prevent a gradual accumulation of dust in either the burner or the air passage, a small cover of the size of the top of B and shaped like the lid of a pill box should be kept upon the lamp when not in use.

The following are the more important dimensions on which the precision of the lamp depends; but no departure should be made from any of the dimensions as shown by the working drawings. All dimensions are given in millimetres.

*Saturator, A.*— $184 \times 184 \times 38$  deep, inside measurement, with seven partitions alternately meeting either side and stopping 25 short of the opposite side to cause the air to pass eight times across the box. These partitions must be soldered to the top, not to the bottom of the box.

*Siphon Tube from Saturator.*—Outer diameter, 14 (half-inch full).

*Indiarubber Tube.*—Inner diameter, 13 (half-inch).

*Steatite Burner.*—Outer diameter, 24; Inner diameter, 14; 30 holes, not less than 1.25 or more than 1.5 in

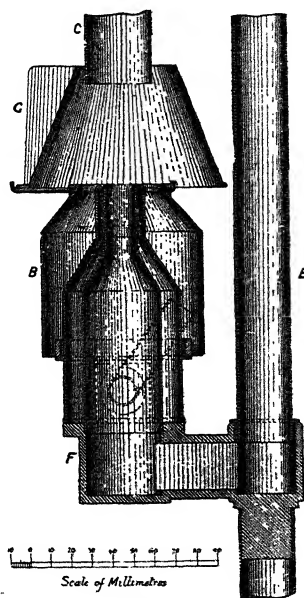


FIG. 2.

diameter. (The holes must be evenly spaced, and in any one burner they must not differ from one another in diameter by more than .05 millimetre.)

*Brass Chimney, c.*—Outer diameter, 32; Inner diameter, 30; Length, 431.

*Brass Outer Tube, d.*—Outer diameter, 52; Inner diameter, 50; Length, 290; Chimney c projects 68 below and 73 above the tube d.

*Brass Tube, e.*—Outer diameter, 25; Inner diameter, 23; Length, 529½; Distance between axis of tube e and axis of tubes c and d, 67.

*Shade, g.*—Diameter of base, 102; Diameter at top, 55; Height, 57; Opening 38 within, 34 without. The structure of the actual burner is shown in the sectional drawing, Fig. 2.

The working drawings are kept at the Gas Referees' office, and may be seen, and notes may be made, after permission in writing has been obtained from the Referees.

*Note.*—The entrance pipe for the pentane, shown dotted in Fig. 2, should be more nearly horizontal, as shown in Fig. 1.

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## APPENDIX B.

The pentane to be used in the 10-candle Lamp should be prepared and tested in the following manner:—

*Preparation.*—Light American petroleum, such as is known as Gasoline and used for making air-gas, is to be further rectified by three distillations, at 55° C., 50°, and 45° in succession. The distillate at 45° is to be shaken up from time to time during two periods of not less than three hours each with one-tenth its bulk of (1) strong sulphuric acid, (2) solution of caustic soda. After these treatments it is to be again distilled, and that portion is to be collected for use which comes over between the temperatures of 25° and 40°. It will consist chiefly of pentane, together with small quantities of lower and higher homologues whose presence does not affect the light of the lamp.

*Testing.*—The density of the liquid pentane at 15° C. should not be less than 0.6235 nor more than 0.626 as compared with that of water of maximum density. The density of the pentane when gaseous, as compared with that of hydrogen at the same temperature and under the same pressure, may be taken. This is done most readily and exactly by Gay Lussac's method, under a pressure of about half an atmosphere and at temperatures

between  $25^{\circ}$  and  $35^{\circ}$ . The density of gaseous pentane should lie between 36 and 38.

Any admixture with pentane of hydrocarbons belonging to other groups and having a higher photogenic value, such as benzene or amylene, must be avoided. Their presence may be detected by the following test. Bring into a stoppered 4-oz. bottle of white glass 10 c.c. of nitric acid, specific gravity 1.32 (made by diluting pure nitric acid with half its bulk of water); add 1 c.c. of a dilute solution of potassium permanganate, containing 0.1 gram of permanganate in 200 c.c. Pour into the bottle 50 c.c. of the sample of pentane, and shake strongly during five successive periods of twenty seconds. If no hydrocarbons other than paraffins are present, the pink colour, though somewhat paler, will still be distinct; if there is an admixture of as much as  $\frac{1}{2}$  per cent. of amylene or benzene, the colour will have disappeared.

#### APPENDIX C.

##### *The Provision of Pentane for use in the Testing Places.*

The following is the procedure which the Gas Referees have arranged with the Gas Companies for the provision and testing of pentane:—

Each of the Gas Companies shall keep upon their premises one or more properly closed vessels capable of containing from fifty to one hundred gallons of pentane, subject to any arrangement by which a Gas Company may obtain their supply of pentane which has been certified by the Gas Referees from another Gas Company.

When a supply of pentane is needed for use in the testing places, a number of metal cans with screw stoppers, of a pattern approved by the Gas Referees, shall be provided sufficient to contain the whole quantity required.

The Gas Referees shall then be informed by letter that this quantity of pentane awaits their examination; and they will arrange to attend at the premises where the pentane is stored. They will see the cans filled, and will affix a numbered lead seal to each can.

They will then take away one or more of the cans for examination; the remaining cans must be kept until the Gas Referees have reported on the quality of the pentane.

If the results of their testings are satisfactory, they will prepare as many labels as there are cans of pentane. Each label will

bear the embossed stamp of the Gas Referees, and will be numbered with the number or numbers impressed upon the lead seals on the cans. These labels will then be sent to the Company for attachment.

No cans of pentane which the Gas Referees have certified are to be supplied to or used by any person or persons other than the Gas Examiners at the several testing places without the written permission of the Gas Referees, and a record must be kept by the Gas Company of the number of cans thus certified from time to time specifying the date and the testing places to which they have been sent. If, however, application should be made to the Gas Referees by the London County Council, the Corporation of London, or any of the Metropolitan Gas Companies, to examine and certify pentane in reasonable quantities for non-official testings, they will be willing to do so.

If the Gas Referees, after examination, find that the sample of pentane taken from any vessel does not satisfy the requirements of their notification, they will inform the Gas Company of the fact; and in such case the lead seals are to be cut off from the other cans filled from the same vessel, and returned to the Gas Referees.

The Gas Companies will send the certified cans of pentane to the testing places in their several districts. The Gas Examiner at any testing place will take the presence of the Gas Referees' lead seal and label, bearing identical numbers, upon any can, as evidence that the pentane therein has been certified, and no pentane shall be used in any testing that has not been so certified.

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## APPENDIX D.

### THE TABLE PHOTOMETER.

The several parts of the apparatus stand upon a well-made and firm table, 5 feet 6 inches by 3 feet 6 inches, and 2 feet 5 inches high. The upper surface of this table is smooth, level, and dead black. Upon this are placed or clamped in the positions shown in Fig. 3:—

1. The Gas Meter.
2. The Gas Governor.
3. The Regulating Tap.
4. The "Metropolitan Argand Burner, No. 2," and Sliding Base.

5. The Flat Flame Burner and Sliding Base.
6. The Slide, Connecting Rod and Photometric Scale, and Index.

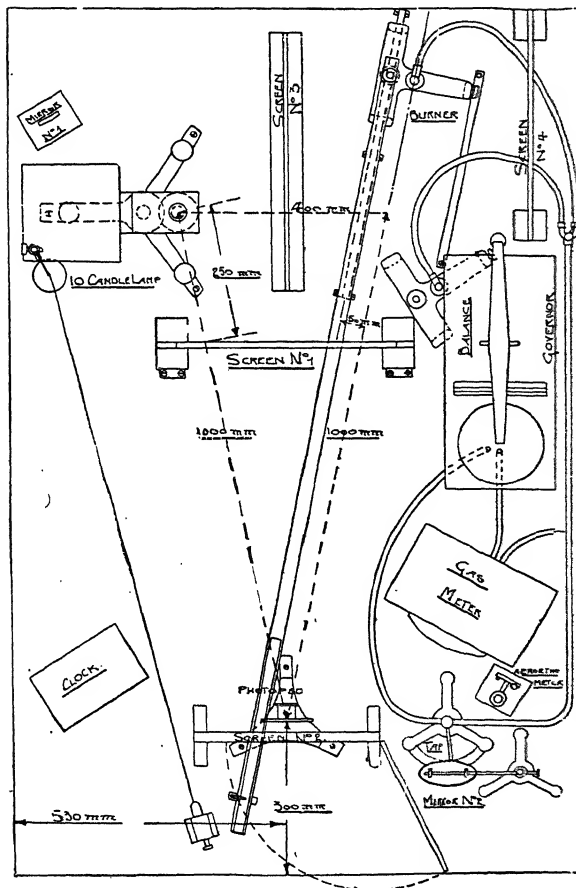


FIG. 3.

7. The Connecting Pipes.
8. The Pentane Ten-Candle Lamp.
9. The Photoped.
10. The Aerorthometer.

11. The Stop-Clock.
12. Dark Screens; Mirrors; Measuring Rod; Small Block, and Pulley.

### 1. *The Gas Meter.*

The Gas Meter is sufficiently described on p. 250 of the Notification.

### 2. *The Gas Governor.*

The Gas Governor must be such as will effectually do away with any variation of pressure produced by the working of the

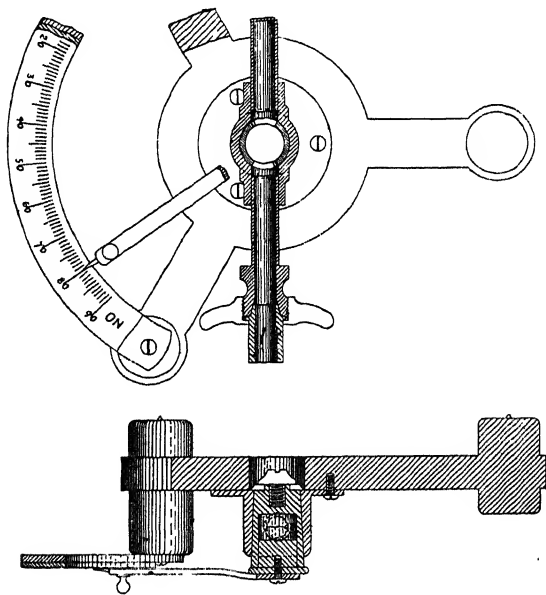


FIG. 4.—THE REGULATING TAP.

meter or other causes. A loose blackened screen,  $8\frac{1}{2}$  inches high by 6 inches wide, should be placed upon the base of the governor near the tank to prevent the water in the tank being heated by the flame of the gas burner.

### 3. *The Regulating Tap.*

This must have a large well-fitting conical plug with a round hole on each side of such a size as to allow gas to pass at the

rate of about 4 cubic feet per hour under the pressure at the outlet of the governor. In addition there must be narrow saw-cuts on opposite sides of the two holes when viewed in plan, which will allow an additional passage of about 2 cubic feet of gas per hour when the tap is so turned that the holes and the saw-cuts are both opposite the orifices of the fixed part of the tap. The construction of the tap is shown in Fig. 4. The

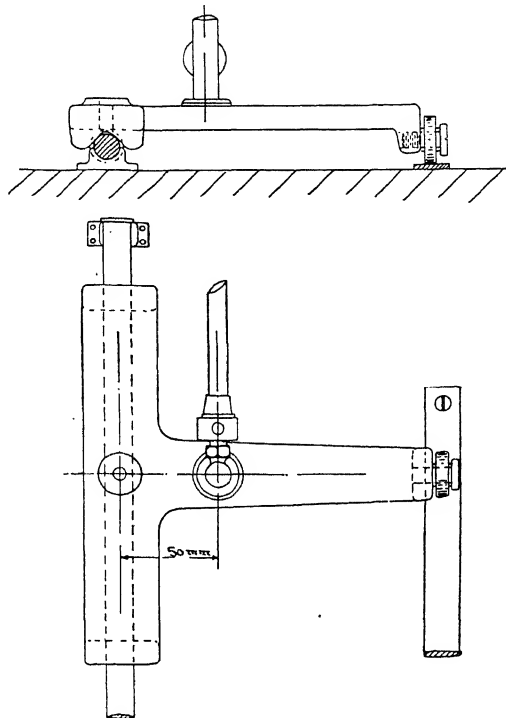


FIG. 5.

index must be secured to the conical plug without any play, and its pointed end must pass over a scale graduated in degrees upon an arc of not less than 80 millimetres radius. The arc is to extend over  $90^\circ$ , and the degrees are to be numbered from  $0^\circ$  to  $90^\circ$ . The arc is to be made of white enamel glass, and the divisions are to be etched upon it, and the marks filled in with black. The tap is to be off when the pointer is at one extremity

of the arc at  $0^\circ$ , and fully on when it is at the other extremity at  $90^\circ$ . The small hole should be fully open at about  $20^\circ$  so that the action of the saw-cuts may extend over the remaining portion of the arc.

The tap must be kept clean and sufficiently lubricated to work easily.

#### 4. *The "Metropolitan Argand Burner No. 2."*

This is the burner described in Appendix E. It is to be mounted upon a tripod capable of moving upon the slides fixed to the table so that its distance from the photoped can be adjusted by means of the connecting rod. The construction of the cast-iron foot and slide is shown in Fig. 5. The height of the top of the cone is 353 millimetres above the table. The axis of the burner should be vertical.

#### 5. *The Flat Flame Burner.*

The flat flame burner is Bray's "No. 7 Economiser" fitted over a Bray's "No. 4 Regulator."

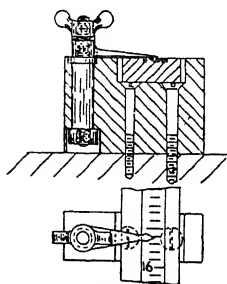


FIG. 6.

The No. 7 Economiser is a slit burner, the width of the slit being .024 inch. The No. 4 Regulator is a union jet burner. Both the economiser and burner contain gauze to steady the flow of gas.

The burner is carried upon a sliding foot of the same construction as that used for the Metropolitan Argand Burner No. 2. An adapter is used to bring the height of the top of the burner to 353 millimetres above the table, and the burner is so turned that the plane of the flame makes an angle of about  $45^\circ$  with the line joining it and the slit in front of the photoped. When the Argand is being used the sliding foot carrying the flat flame burner may be made to stand out of the way as shown in Fig. 3. When the flat flame burner is being used the sliding foot carrying it is placed upon the slide and the Argand is pushed back as far as it will go, or if necessary it may be lifted off the slide. In either case the chimney should be removed so as not to reflect light upon the photoped.

#### 6. *The Slide, Connecting Rod, and Photometric Scale.*

The slide fixed to the table on which the bases of the burners move constitutes with either of them a geometric slide, there being



the necessary five independent points of support. A rod of drawn brass, half an inch in diameter, is supported by three feet screwed to the table so that its under surface is one-eighth of an inch above the table. This rod is screwed down so as to be 50 millimetres to the left of the line, shown dotted in Fig. 3, which joins the gas burner with the slit in front of the photoped. Parallel with this, and at such a distance as to allow the wheel on the foot to run upon it, is screwed a piece of flat rolled brass, as shown. Exactly 50 millimetres from the centre of the upright gas pipe, and in a direction at right angles to the line of motion, a hole,  $\frac{1}{4}$  inch in diameter, is made in the foot of each burner, into which a slightly tapered pin near one end of the connecting rod is to be placed, so that the burner may be pulled on the slide by the observer. This connecting rod is supported near the other end, as shown in Fig. 3, by a block, so placed that the rod is parallel with the dotted line already mentioned. The details of this block are shown in Fig. 6. The connecting rod has let into it at the observer's end a strip of white enamel glass, on which is etched a photometric scale extending from 8 to 17 candles, and divided into tenths of a candle. A portion of this scale and the pattern to be followed is shown in Fig. 7. The index of the block is adjustable, so that when the Argand burner is placed at the 10 candle distance by means of the measuring rod, as described under (12), the index may be clamped over the division 10. Then at all distances the number read upon the scale will accurately represent the candle-power of the gas flame.

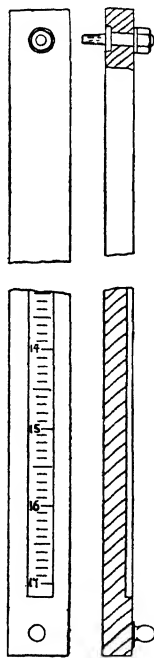


FIG. 7.

#### 7. *The Connecting Pipes.*

These are to be made of half-inch (outside measure) composition piping except in the case of the two pipes which connect the three-way tap on the table with the two burners, where flexible metallic tube is to be used. They are to be connected with the different pieces of apparatus by  $\frac{3}{8}$ -inch unions, except in the case of the gas meters, where the unions belonging to the meter may be retained. In all cases the boss of the union is to be attached to the apparatus and the cap and lining to the ends of the connecting pipe. These pipes are to be placed above the table. No grooves, recesses, or holes, other than the screw holes for the screws referred to in this Appendix, are to be made in the table.

### 8. *The Ten-Candle Pentane Lamp.*

This is described in Appendix A.

The lamp is placed in position upon the table, the exact distance of the steatite ring from the photoped being determined by means of the measuring rod as described under (12), p. 265. The swivel feet, which should fit loosely on the screws, shall then be clamped in position by the aid of the clamps shown in Fig. 8. The height of the top of the steatite burner is 353 millimetres above the table.

### 9. *The Photoped.*

The photoped is represented in Fig. 9; it consists of the following parts: a plate, 100 millimetres square, with a central hole, 21 millimetres square. This is held in a vertical position by an up-

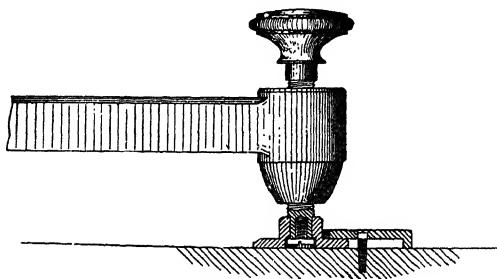


FIG. 8.

right support so that the centre of the square is 400 millimetres above the table. The upright is carried by a tripod with flat feet clamped to the table by clamps as shown in Fig. 9. To one face of the square plate is fastened, by two binding screws, a clamping plate, 60 × 40 millimetres, also with a central hole, 21 millimetres square, so that the two openings are opposite one another. A piece of suitable white paper is pinched between the two plates so as to cover the openings and project a little way below the clamping plate. To the upper surface of the square plate is fixed a strip of glass, so that the lower edge is close to, and exactly parallel to, the plate, while the upper edge is so much in advance as will allow the reflection of the flames described on p. 267 to be observed. The clamping plate carries centrally a horizontal tube about 35 millimetres in diameter and 30 in length. In this slides smoothly a tube containing a diaphragm in which a rectangular slit, 1 millimetres, has been cut.

10. *The Aerorthometer.*

The Aerorthometer is described and illustrated in Appendix F.

In using the Aerorthometer, turn the screw up until the level of the mercury in the open tube is some distance below that of the mercury in the bulb tube; then turn the screw slowly down until the mercury stands at the same level in both tubes. The division at which the mercury now stands is the Aerorthometer reading.

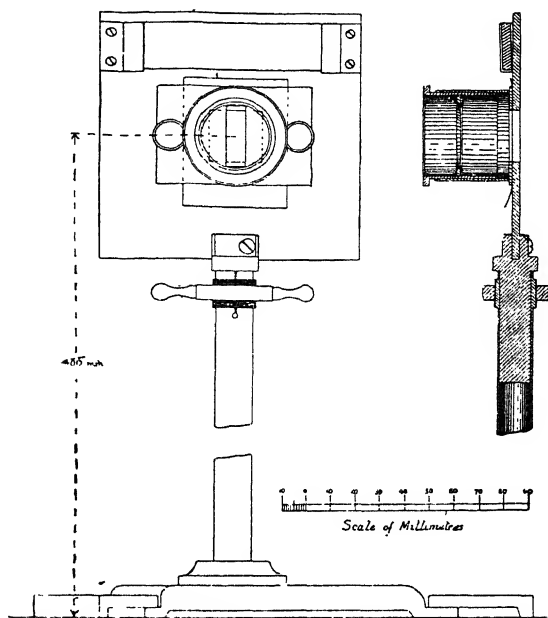


FIG. 9.

The Gas Examiner shall, not less often than once a month, compare the Aerorthometer reading with the reciprocal of the Tabular Number deduced from observations of Barometer and Thermometer, and if there is a difference of more than one-half per cent. the Aerorthometer is to be readjusted. If at any time the Aerorthometer is out of order the reciprocal of the tabular number is to be used.

### 11. *The Stop-Clock.*

This is the clock ordinarily used in testing places, either attached to the meter or independent of it. It must be provided with mechanism for starting and stopping. It will facilitate the comparison with the standard clock if it is made to give an audible sound, by means of a bell or otherwise, at the completion of each minute, and the number of minutes up to ten at least should be indicated by a separate hand.

### 12. *Dark Screens; Mirrors; Measuring Rod.*

Five dark screens are provided in order to prevent the inaccuracy and inconvenience to which stray light would give rise.

The first is placed between the burners and the photoped in the position shown in Fig. 3. This screen is 500 millimetres wide and 400 millimetres high with its lower edge 100 millimetres above the table. It has two rectangular openings. The opening to the left is 40 millimetres wide and 55 high, and its lower edge is 350 millimetres above the table. The opening to the right is 75 millimetres wide, its lower edge is 340 millimetres above the table, and it extends to the top of the screen. The centre lines of these two openings are 300 millimetres apart. The screen is carried by two feet hinged to the table as shown in Fig. 3. Care must be taken that it is so placed that the whole of the flame under the tube of the 10-candle lamp and the whole of the chimney and burner of the Argand or the whole of the flat flame can be seen through all parts of the slit in front of the photoped when the paper is removed for that purpose.

The second dark screen consists of a piece of black velvet or black cloth 350 millimetres square stretched on a frame and supported so that its lower edge is 150 millimetres above the table. In this is cut a hole 50 millimetres square with its lower edge 380 millimetres above the table. This screen is placed close to the photoped but on the opposite side to that facing the lamps, and with the square hole opposite the square hole in the plate of the photoped. To the right side of the frame is hinged a light frame 350 millimetres high and 300 wide, with its lower edge 150 millimetres above the table. On this also is stretched black velvet or black cloth. This prevents the illuminated dial of the meter or arc of the regulating tap from interfering with the photometric observations, while at the same time it can be readily moved when these are to be observed.

The third dark screen is about 500 millimetres wide and 570 high. The fourth is about 450 wide and 570 high. These may be made of card painted dead black, or of thin wood, and may be

placed approximately in the positions shown in Fig. 3 and with their lower edges 180 millimetres above the table. In the fourth screen there is an opening about 150 millimetres square, fitted with glass of neutral tint, through which the height of the flame may be observed without fatiguing the eye.

The fifth dark screen consists of a piece of black velvet or cloth large enough to form a black background to the lamps when viewed from the photoped. It is best placed upon the wall, but if that is inconvenient or other objects intervene, it should be supported on a stand, but always so as to be at least 300 millimetres behind the flames of the photometer.

Three small mirrors are carried on light stands. One of these is made of ordinary flat silvered glass and is so placed as to enable the Gas Examiner, when seated at the photoped end of the table, on moving his head to the left of the second dark screen, to see by reflection the tip of the flame of the 10-candle lamp through the mica window in the tube c.

The second, which should be about 120 millimetres in diameter, is convex, and should have a radius of curvature of about 400 millimetres. It is placed on the observer's right, and is so inclined that it casts a divergent beam of subdued light upon the divided arc of the regulating tap, upon the face of the meter, upon the aerorthometer, and upon the Gas Examiner's note-book.

The third, which is of flat glass, is placed on the top of the second screen, and is so arranged as to throw light on the photometric scale.

All the apparatus on the table upon which light can fall and which might by reflection illuminate the photoped, or catch the eye of the operator, is to be painted dead black; or, if of finished brass, it is to be bronzed before being lacquered.

The correct position of the photoped and of the burners is to be verified as follows:—A measuring rod has securely fastened to it transversely a cylindrical and shouldered plug which just fits into the steatite rings of the 10-candle lamp and of the Metropolitan Argand, there being a step on the plug to enable it to be used for either. The rod is balanced about and rests upon the burner. The rod is to be 1,000 millimetres from the axis of the plug to the extremity of the rounded ivory point. The rod must be capable of being placed in either burner without disarranging it, except in the removal of the glass chimney of the Metropolitan Argand or the conical shade of the 10-candle lamp. When the rod is in position upon either burner and the long end is moved gradually round toward the photoped, it should just come in contact with the paper under the clamping plate at the middle point.



When the burners have been lighted and the flames turned low, the reflection of one is to be observed over the other in the glass of the photoped. It should appear central ; in that case the photoped is symmetrically placed with respect to the two burners. If the reflection does not appear central, the nut on the standard is to be loosened, and the plate turned until the reflection is central. The two lights are then to be turned up and the slit is to be moved in or out until the two rectangular spaces illuminated by the two lights just meet but do not overlap.

A pattern Table Photometer is set up in the Gas Referees' Office. This may be seen after permission in writing has been obtained from the Referees.

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## APPENDIX E.

The burner which has been adopted as the Standard Burner for testing gas was devised by Mr. Charles Carpenter, and has been called by him "The Metropolitan Argand Burner No. 2."

A full-sized drawing showing details is given in Fig. 10, on which also are marked the important dimensions. While these are given in every case to the nearest thousandth of an inch, this degree of accuracy is not essential. The important dimensions are those governing the gas and air passages, but all should be adhered to as nearly as workshop practice allows.

The annular chamber from which the gas issues is made of steatite.

The chimney to be used with this burner is 6 inches long and  $1\frac{5}{8}$  inch in internal diameter.

Each testing place is provided with a box containing two wire gauges, one 0.058 inch, and the other 0.062 inch in diameter. The Gas Examiner must once in every month pass the smaller gauge through every hole in the burner, so as to clear out any loose obstruction or detect any hard concretion that might interfere with the proper discharge of the gas. He should at the same time satisfy himself that the larger gauge will not pass through the holes.

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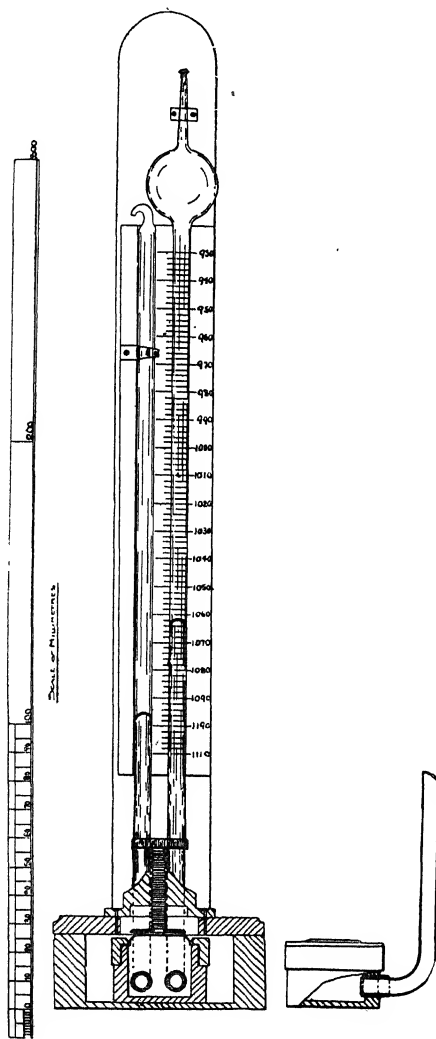


FIG. II.



## APPENDIX F.

## THE AERORTHOMETER.

This is illustrated in Fig. 11. The mode of reading the instrument has been explained, p. 263 (10). A reading furnishes the figure required for correcting the volume of a gas measured over water at any ordinary temperature and pressure to that which the gas would have if measured over water under a pressure of 30 inches of mercury and at a temperature of 60° Fahr. Thus its reading corresponds to the figure derivable from a reading of the barometer and the thermometer and a reference to a table giving the tension of aqueous vapour at different temperatures. The instrument consists of a bulb and vertical stem in which sufficient water is present to ensure that the air is saturated. The measuring tube, which terminates in a closed bulb, and a companion tube of the same calibre which is open to the air, dip into a reservoir of mercury in the base, the capacity of which can be adjusted by a regulating screw pressing on a leather cover. The relative volume of the bulb and tube down to any division is represented by the number belonging to that division. The capillary tube above the bulb is closed by a very small amount of sealing wax. In order to adjust the instrument the sealing wax is softened by heat and a small hole made through it. When the bulb has acquired the temperature of the air the regulating screw is to be turned until the two columns of mercury stand level at the calculated Aerorthometer reading. Then the sealing wax stopping is again melted where it was perforated, by being touched from above with a heated wire while the base of the tube and the bulb are protected from heat by a wrapping of cotton wool.

## APPENDIX G.

TABULAR NUMBERS, BEING A TABLE TO FACILITATE THE  
AT DIFFERENT TEMPERATURES AND UNDER

BAR.	THERMOMETER—FAHRENHEIT.										
	40°	42°	44°	46°	48°	50°	52°	54°	56°	58°	60°
28.0	.979	.974	.970	.965	.960	.956	.951	.946	.942	.937	.932
28.1	.983	.978	.973	.969	.964	.959	.955	.951	.945	.941	.936
28.2	.986	.981	.977	.972	.967	.963	.958	.953	.949	.944	.939
28.3	.990	.985	.980	.976	.971	.966	.961	.957	.952	.947	.942
28.4	.993	.988	.984	.979	.974	.970	.965	.960	.955	.951	.946
28.5	.997	.992	.987	.983	.978	.973	.968	.964	.959	.954	.949
28.6	1.001	.995	.991	.986	.981	.977	.972	.967	.962	.958	.953
28.7	1.004	.999	.994	.990	.985	.980	.975	.970	.966	.961	.956
28.8	1.007	1.003	.998	.993	.988	.984	.979	.974	.969	.964	.959
28.9	1.011	1.006	1.001	.997	.992	.987	.982	.977	.973	.968	.963
29.0	1.014	1.010	1.005	1.000	.995	.990	.986	.981	.976	.971	.966
29.1	1.018	1.013	1.008	1.004	.999	.994	.989	.984	.979	.975	.969
29.2	1.021	1.017	1.012	1.007	1.002	.997	.992	.988	.982	.978	.973
29.3	1.025	1.020	1.015	1.011	1.006	1.001	.996	.991	.986	.981	.976
29.4	1.028	1.024	1.019	1.014	1.009	1.004	.999	.995	.990	.985	.980
29.5	1.032	1.027	1.022	1.018	1.013	1.008	1.003	.998	.993	.988	.983
29.6	1.036	1.031	1.026	1.021	1.016	1.011	1.006	1.001	.996	.992	.986
29.7	1.039	1.034	1.029	1.025	1.019	1.015	1.010	1.005	1.000	.995	.990
29.8	1.043	1.038	1.033	1.028	1.023	1.018	1.013	1.008	1.003	.998	.993
29.9	1.046	1.041	1.036	1.031	1.026	1.022	1.017	1.012	1.007	1.002	.997
30.0	1.050	1.045	1.040	1.035	1.030	1.025	1.020	1.015	1.010	1.005	1.000
30.1	1.053	1.048	1.043	1.038	1.033	1.029	1.024	1.019	1.014	1.009	1.003
30.2	1.057	1.052	1.047	1.042	1.037	1.032	1.027	1.022	1.017	1.012	1.007
30.3	1.060	1.055	1.050	1.045	1.040	1.036	1.030	1.025	1.020	1.015	1.010
30.4	1.064	1.059	1.054	1.049	1.044	1.039	1.034	1.029	1.024	1.019	1.014
30.5	1.067	1.062	1.057	1.052	1.047	1.042	1.037	1.032	1.027	1.022	1.017
30.6	1.071	1.066	1.061	1.056	1.051	1.046	1.041	1.036	1.031	1.026	1.020
30.7	1.074	1.069	1.064	1.059	1.054	1.049	1.044	1.039	1.034	1.029	1.024
30.8	1.078	1.073	1.068	1.063	1.058	1.053	1.048	1.043	1.037	1.032	1.027
30.9	1.081	1.076	1.071	1.066	1.061	1.056	1.051	1.046	1.041	1.036	1.031
31.0	1.085	1.080	1.075	1.070	1.065	1.060	1.055	1.049	1.044	1.039	1.034

Values in the above table have been calculated from the formula  $v = \frac{17.64 (h - a)}{460 + t}$ ,  
and  $a$  the tension of aqueous vapour at  $t^\circ$ . If  $v$  is any volume at  $t^\circ$  and  $h$  inches

## APPENDIX G.

CORRECTION OF THE VOLUME OF GAS MEASURED OVER WATER  
DIFFERENT ATMOSPHERIC PRESSURES.

THERMOMETER—FAHRENHEIT.											
62°	64°	66°	68°	70°	72°	74°	76°	78°	80°	82°	84°
.927	.922	.917	.912	.907	.902	.897	.892	.887	.881	.875	.870
.930	.926	.921	.916	.911	.905	.900	.895	.890	.884	.879	.873
.934	.929	.924	.919	.914	.909	.904	.898	.893	.887	.882	.876
.937	.932	.928	.922	.917	.912	.907	.902	.896	.891	.885	.880
.941	.936	.931	.926	.921	.915	.910	.905	.900	.894	.888	.883
.944	.939	.934	.929	.924	.919	.914	.908	.903	.897	.892	.886
.947	.943	.938	.932	.927	.922	.917	.912	.906	.901	.895	.889
.951	.946	.941	.936	.931	.925	.920	.915	.909	.904	.898	.893
.954	.949	.944	.939	.934	.929	.924	.918	.913	.907	.901	.896
.958	.953	.948	.942	.937	.932	.927	.921	.916	.910	.905	.899
.961	.956	.951	.946	.941	.935	.930	.925	.919	.914	.908	.903
.964	.959	.954	.949	.944	.939	.933	.928	.923	.917	.911	.906
.968	.963	.958	.952	.947	.942	.937	.931	.926	.920	.914	.909
.971	.966	.961	.956	.950	.945	.940	.935	.929	.923	.918	.912
.975	.969	.964	.959	.954	.949	.943	.938	.932	.927	.921	.915
.978	.973	.968	.962	.957	.952	.947	.941	.936	.930	.924	.919
.981	.976	.971	.966	.960	.955	.950	.944	.939	.933	.927	.922
.985	.980	.974	.969	.964	.959	.953	.948	.942	.937	.931	.925
.988	.983	.978	.972	.967	.962	.957	.951	.946	.940	.934	.928
.991	.986	.981	.976	.970	.965	.960	.954	.949	.943	.937	.932
.995	.990	.985	.979	.974	.968	.963	.958	.952	.946	.941	.935
.998	.993	.988	.983	.977	.972	.966	.961	.955	.950	.944	.938
1.002	.996	.991	.986	.980	.975	.970	.964	.959	.953	.947	.941
1.005	1.000	.995	.989	.984	.978	.973	.968	.962	.956	.950	.945
1.008	1.003	.998	.993	.987	.982	.976	.971	.965	.959	.954	.948
1.012	1.006	1.001	.996	.990	.985	.980	.974	.969	.963	.957	.951
1.015	1.010	1.005	.999	.994	.988	.983	.977	.972	.966	.960	.954
1.018	1.013	1.008	1.003	.997	.992	.986	.981	.975	.969	.963	.957
1.022	1.017	1.011	1.006	1.000	.995	.990	.984	.978	.972	.967	.961
1.025	1.020	1.015	1.009	1.004	.998	.993	.987	.982	.976	.970	.964
1.029	1.023	1.018	1.013	1.007	1.002	.996	.991	.985	.979	.973	.967

where  $h$  is the height of the barometer in inches,  $t$  the temperature on the Fahrenheit scale, pressure and  $V$  the corresponding volume at 60° and 30 inches pressure,  $V = v n$ .

## APPENDIX H.

## TEST FOR SULPHURETTED HYDROGEN.

The apparatus represented by Fig. 12 consists of a plate with a circular channel half filled with mercury in which rests a bell-glass, held down in position by an arm and cap not shown in the figure. A central tube connected below with the gas inlet rises nearly to the top of the bell-glass, and carries midway wires pointed and curved at the end, from each of which a slip of lead paper hangs.

A second pipe passing through the plate and terminating above

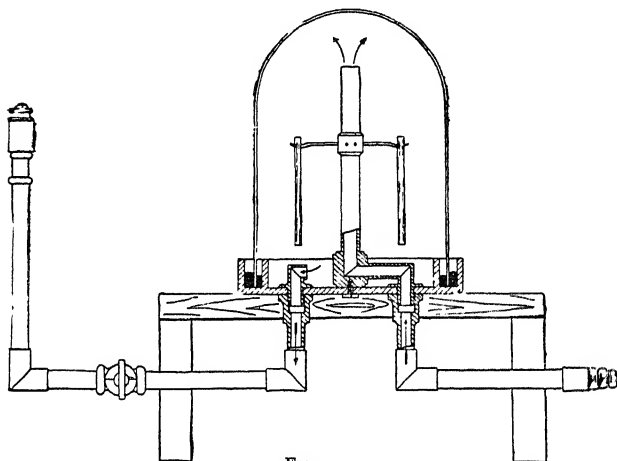


FIG. 12.

in a short elbow provides an outlet for the gas, which is burnt as it issues from a governor-burner passing gas at about the rate of five cubic feet per hour.

## APPENDIX K.

## SULPHUR TEST.

The apparatus to be employed is represented by Fig. 13, and the following description:—The gas is burnt in a small Burner with a steatite top, which is mounted on a short cylinder, perforated with holes for the admission of air, and

having on its upper surface, which is also perforated, a deep circular channel to receive the wide end of a glass trumpet-tube. There are both in the side and in the top of this stand fourteen holes of 5 millimetres in diameter, or an equivalent air-way. On the top of the stand, between the narrow stem of the burner and the surrounding glass trumpet-tube, are to be placed pieces of commercial sesqui-carbonate of ammonia weighing in all about 2 ounces.

The products both of the combustion of the gas and of the gradual volatilisation of the ammonia salt go upwards through the trumpet-tube into a vertical glass cylinder with a tubulure near the bottom, and drawn in at a point above this to about half its diameter. From the contracted part to the top the cylinder is packed with balls of glass about 15 millimetres in diameter, to break up the current and promote condensation. From the top of this condenser there proceeds a long glass pipe or chimney slightly bent over at the upper end, serving to effect some further condensation, as well as to regulate the draught and afford an exit for the uncondensable gases. In the bottom of the condenser is fixed a small glass tube, through which the liquid formed during the testing drops into a flask placed beneath.

The following cautions are to be observed in selecting and setting up the apparatus:—

See that the inlet pipe fits gas-tight into the burner, and that the holes in the circular stand are clear. If the burner gives a luminous flame, remove the top piece, and having hammered down gently the nozzle of soft metal, perforate it afresh, making as small a hole as will give passage to two-thirds of a cubic foot of gas per hour at a convenient pressure.

See that the tubulure of the condenser has an internal diameter of not less than 18 millimetres, and that its outside is smooth and of the same size as the small end of the trumpet-tube; also that the internal diameter of the contracted part is not less than 30 millimetres.

See that the short piece of indiarubber pipe fits tightly both to the trumpet-tube and to the tubulure of the condenser.

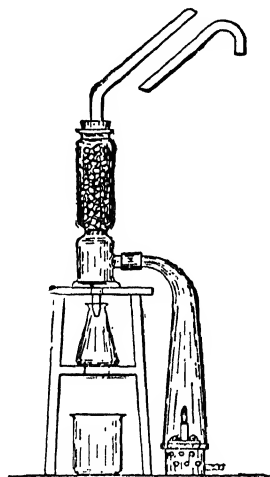


FIG. 13.

The small tube at the bottom of the condenser should have its lower end contracted, so that when in use it may be closed by a drop of water.

The indiarubber pipe at the lower end of the chimney-tube should fit into or over, and not simply rest upon, the mouth of the condenser.

A central hole, about 50 millimetres in diameter, may with advantage be made in the shelf of the stand. If a beaker is kept on the table below, the liquid will still be preserved if by any accident the flask is not in its place.

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## APPENDIX L.

### THE GAS CALORIMETER.

The Gas Calorimeter, which has been designed by Mr Boys, is shown in vertical section in Fig. 14. It consists of three parts, which may be separated, or which, if in position, may be turned relatively to one another about their common axis. The parts are (1) the base A, carrying a pair of burners B, and a regulating tap. The upper surface of the base is covered with a bright metal plate held in place by three centering and lifting blocks C. The blocks are so placed as to carry (2) the vessel D which is provided with a central copper chimney E and a condensed water outlet F. Resting upon the rim of the vessel D are (3) the water circulating system of the calorimeter attached to the lid G. Beginning at the centre where the outflow is situated there is a brass box which acts as a temperature equalising chamber for the outlet water. Two dished plates of thin brass K K are held in place by three scrolls of thin brass L L L. These are simply strips bent round like unwound clock springs, so as to guide the water in a spiral direction inwards, then outwards and then inwards again to the outlet. The lower or pendent portion of this box is kept cool by circulating water, the channel for which may be made in the solid metal, as shown, on the right side, or by sweating on a tube as shown on the left. Connected to the water channel at the lowest point by a union are five or six turns of copper pipe such as is used in a motor car radiator of the kind known as Clarkson's. In this a helix of copper wire threaded with copper wire is wound round the tube, and the whole is sweated together by immersion in a bath of melted solder. A second coil of pipe of similar construction surrounding the first is fastened to it at the lower end by a union. This terminates at the upper end in a block, to which the inlet water box and thermometer holder are secured by a union as shown

at o. An outlet water box P and thermometer holder are similarly secured above the equalising chamber H. The lowest turns

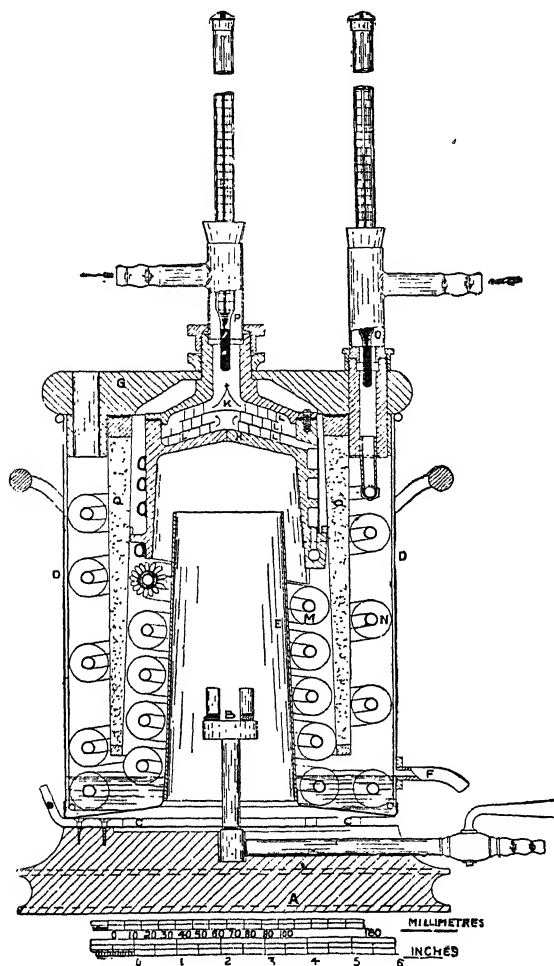


FIG. 14.

of the two coils M'N are immersed in the water which in the first instance is put into the vessel D.

Between the outer and inner coils  $MN$  is placed a brattice  $Q$  made of thin sheet brass, containing cork dust to act as a heat insulator. The upper annular space in the brattice is closed by a wooden ring, and that end is immersed in melted rosin and bees-wax cement to protect it from any moisture which might condense upon it. The brattice is carried by an internal flange which rests upon the lower edge of the casting  $H$ . A cylindrical wall of thin sheet brass, a very little smaller than the vessel  $D$ , is secured to the lid so that when the instrument is lifted out of the vessel and placed upon the table the coils are protected from injury. The

narrow air space between this and the vessel  $D$  also serves to prevent interchange of heat between the calorimeter and the air of the room.

The two thermometers for reading the water temperatures and a third for reading the temperature of the outlet air are all near together and at the same level. The lid may be turned round into any position relatively to the gas inlet and condensed water drip that may be convenient for observation, and the inlet and outlet water boxes may themselves be turned so that their branch tubes point in any direction.

A regular supply of water is maintained by connecting one of the two outer pipes of the overflow funnel shown in Fig. 15 to a small tap over the sink. The overflow funnel is fastened to the wall about 1 metre above the sink and the other outer pipe is connected to a tube in which

there is a diaphragm with a hole about 2.3 mm. in diameter. This tube is connected to the inlet pipe of the calorimeter. A piece of stiff rubber pipe long enough to carry the outflow water clear of the calorimeter is slipped on to the outflow branch and the water is turned on so that a little escapes by the middle pipe of the overflow funnel, and is led by a third piece of tube into the sink. The amount of water that passes through the calorimeter in four minutes should be sufficient to fill the graduated vessel shown in Fig. 16 to some point above the division, but insufficient in five minutes to come above the next division. If this is not found to be the case, a moderate

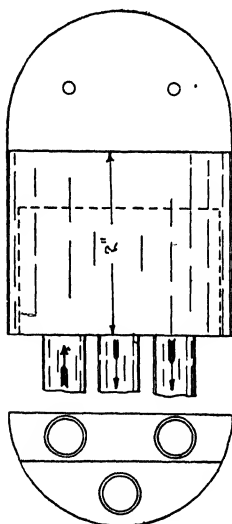


FIG. 15.



lowering of the overflow funnel or reaming out of the hole in the diaphragm will make it so. The overflow funnel should be provided with a lid to keep out dust.

The thermometers for reading the temperature of the inlet and outlet water should be divided on the Centigrade scale into tenths of a degree, and they should be provided with reading lenses and pointers, such as are shown in Fig. 17, that will slide upon them. The thermometers are held in place by corks fitting the inlet and outlet water boxes. The positions of these thermometers should be interchanged every month. The thermometers for reading the temperature of the air near the instrument and of the effluent gas should be divided on the Centigrade scale into degrees.

The flow of air to the burners is determined by the degree to which the passage is restricted at the inlet and at the outlet. The blocks *c* which determine the restriction at the inlet are made of metal  $\frac{3}{16}$  inch or about 5 millimetres thick, while the holes round the lid which determine the restriction at the outlet are five in number and are  $\frac{5}{8}$  inch or 16 millimetres in diameter. The thermometer used for finding the temperature of the effluent gas is held by a cork in the sixth hole in the lid so that the bulb is just above the upper coil of pipe.

The calorimeter should stand on a table by the side of a sink so that the condensed water and hot-water outlets overhang and

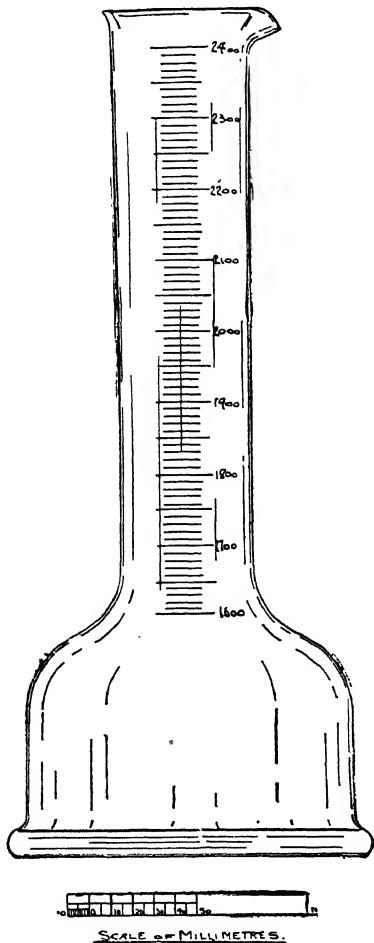


FIG. 16.

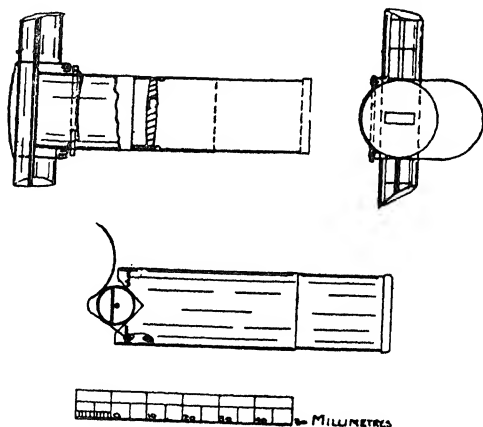


FIG. 17.

should be taken that the small side hole is not covered by the tube. A glass vessel must be provided of the size of the vessel D containing water in which is dissolved sufficient carbonate of soda to make it definitely alkaline. The calorimeter after use is to be lifted out of its vessel D and placed in the alkaline solution and there left until it is again required for use. The liquid should not, when the

deliver into the sink. A suitable change-over funnel is shown in Fig. 18. A piece of indiarubber tube reaching nearly to the base should be attached to the waste waterpipe so as to avoid splashing, and another piece may conveniently be slipped on to the condensed water outlet so as to lead the condensed water into a flask, but care

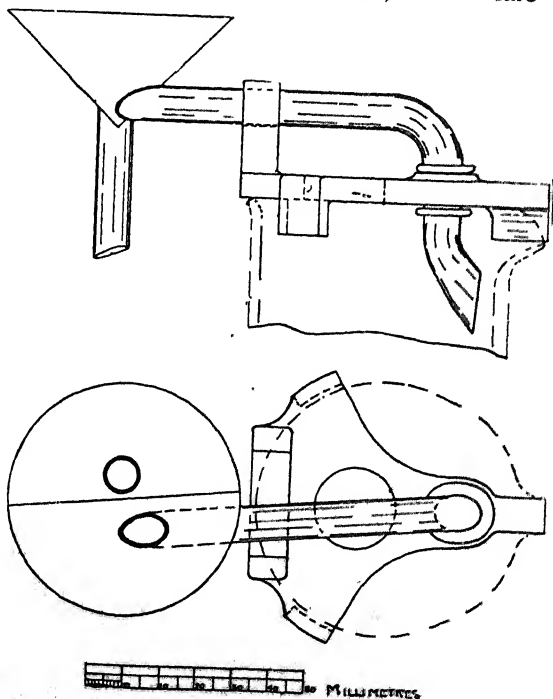


FIG. 18.

calorimeter is placed in it, come within 2 inches of the top of the vessel. The liquid must be replenished from time to time, and its alkalinity must be maintained.

CALORIFIC POWER OF GAS.

FORM WITH EXAMPLE OF CALCULATION (see p. 249).

Water.		Air.		Time by Stop-Clock.
Inlet.	Outlet.	Inlet.	Outlet.	
8.45° C.	33.22° C.	15° C.	12° C.	4 min. 2 sec. = 242 sec.
	.23	One-sixth difference = 0.5.		
	.23			
8.46	.23	Barometer, 29.9 inches ... }		
	.21	Meter thermometer, 60° F. }		
	.22			
	.23			
	.23	Water collected, 2.080 litres.		
8.46	.23	Condensed water in 20 min. = 1,200 sec., 40.3 c.c.		
	.22			
	.21			
	.23			
8.47	.24	Log.	24.77	= 1.3939
8.46	.24	Log.	3	= .4771
	.24	Log.	2.080	= 3181
				<u>2.1801</u>
	5)3.41			
	3) .682	Log.	.997	= 1.9987
	33.23	Log.	155.0	= 2.1904
	8.46	Subtract 0.5		
	<u>24.77</u>			
		<u>154.5</u>	=	Gross Calorific Power.
Log.	40.3	=	1.605	Log. 1200 = 3.079
Log.	242	=	2.384	Log. .997 = 1.999
Log.	1.86	=	.270	<u>3.078</u>
Gross	154.5		<u>4.259</u>	
Log.	15.2	=	<u>1.181</u>	
	<u>139.3</u>	=	Net Calorific Power.	

## APPENDIX M.

## THE GAS REFEREES' STREET LAMP PRESSURE GAUGE.

This instrument is for the purpose of testing in any street at any hour the pressure at which gas is supplied. Its construction and mode of use are as follows:—

Within a lantern, provided with a handle for carrying and feet for resting on the ground, is placed a candle-lamp, to give light for reading the gauge. In front of the candle-lamp is a sheet of opal glass, and in front of this a glass U tube, partly filled with coloured water, and communicating at one end with the air, at the other with a metal pipe, which passes through the bottom of the lantern. In order to read easily and accurately the difference of level of the liquid in the two limbs, a scale divided into tenths of an inch is made to slide between them with sufficient friction to retain it in any position. The zero of the scale having been brought level with the surface of the liquid which is exposed to the gas, the height above this of the surface which is exposed to the air can be read directly. The lantern is closed in front by a glass door, at each side of which is a reflector for throwing light upon the scale of the gauge. Above each limb of the U tube is a tap which can be closed when the instrument is not in use, to prevent the liquid being accidentally spilt.

To make a testing of pressure the governor and burner of a street lamp are to be removed, and the pressure gauge is to be screwed on to the gas-pipe, by which it is supported. In places where incandescent burners are used, the L-shaped pipe described on p. 249 is to be used for the attachment of the pressure gauge. The cock is then turned on, and a reading made.

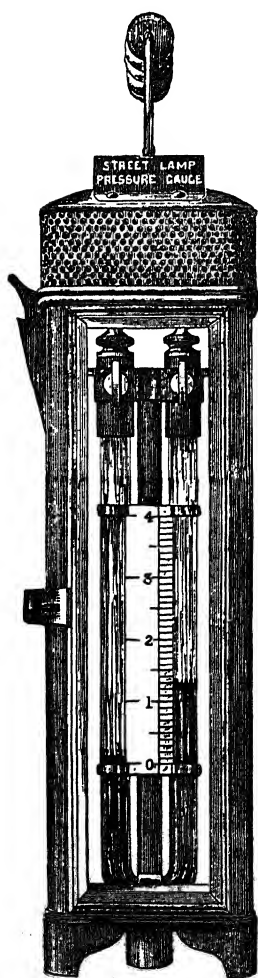


FIG. 19.

If on turning off the cock the level of the liquid is unchanged, or changes slowly, the reading is correct; but if the level changes quickly, the junction between the lamp and the gauge must be made more perfect, and the testing repeated. A small leakage is immaterial, provided the cock is turned fully on.

The pressure at the top of a lamp column is greater by about 0.1 inch than that at the main, which is the pressure required. Accordingly a deduction of 0.1 inch from the observed pressure is to be made.

#### APPENDIX N.

##### THE GAS REFEREES' ONE-TWELFTH OF A CUBIC FOOT MEASURE.

This instrument, which was designed by Mr Harcourt, is represented in Fig. 20; it consists of a vessel of blown glass of a cylindrical form with rounded ends terminating in short tubes about 40 millimetres in diameter outside, which tubes are reduced at their outer ends to about 20 millimetres in diameter outside. Lines are etched round each tubular neck in such positions that the capacity of that portion of the vessel included between these marks is exactly one-twelfth of a cubic foot when the glass is at the ordinary temperature. No correction is needed for the cubical expansion of the glass. The two tubular necks of the instrument pass through two boards placed below and parallel to the top of a small four-legged table. For convenience the upper one of these two boards is made in two parts and hinged to the legs.

Into each end of the instrument a glass tube about 8 millimetres in diameter outside is fitted gas and water-tight by means of india-rubber corks, in such positions that the inner end of the upper tube lies exactly in the plane of the mark at its end of the instrument, while that of the lower is about 1 mm. below the mark.

The upper tube terminates in a T, each branch of which is provided with a stop-cock.

A separate stand carries two shelves, the upper one about 40 millimetres below the level of the upper mark and the lower one below the level of the lower mark. The lower shelf is adjustable, and must be so placed that the action about to be described shall take place.

A water vessel is provided having a capacity of about one-tenth of a cubic foot. It should be made of brass or copper, tinned on the inside. It has a tubulure near the bottom, to which is fitted a metal tap. The end of the tap is to be turned slightly downwards,

and is to have a diameter outside of about 8 millimetres. The size of the way through the tap and of the connections is such that when a meter is being proved in the manner to be described, the water fills the instrument from one mark to the other in about one minute. The water vessel has a tubulure above for filling it, closed by a cork through which passes a narrow glass tube, so that

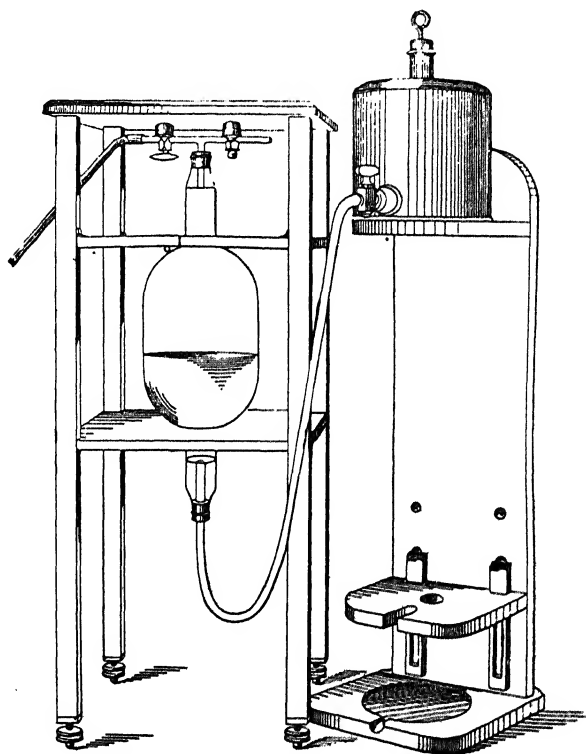


FIG. 20.

air may enter or escape. The end of the tube is bent round upon itself in the form of a crook, so as to exclude dust and dirt. An indiarubber tube connects the tube at the base of the measure with the stop-cock of the water vessel. An ordinary chemical thermometer is provided for taking the temperature of the water.

The pipe supplying gas to each meter is provided near the

meter with a three-way stop-cock carrying a short branch pipe, so formed that it either connects the gas supply only with the branch pipe, the meter only with the branch pipe, or the gas supply with the meter, in which latter case the branch pipe is cut off from both. The index of the tap shows which communication is open. The branch pipe is so shaped as to be convenient for the attachment of an indiarubber tube.

In order to put the instrument in adjustment the water vessel is placed upon the upper shelf, and water is poured into it until the water has risen about one-quarter of an inch in the upper narrow tube of the glass measure. One branch of the glass T is then connected by an indiarubber pipe with the branch of the three-way stop-cock. This is now turned so as to connect the branch pipe with the gas supply. The stop-cock in the branch of the glass T to which the rubber tube is attached is turned on, and the water vessel is placed on the lower self. The water will run back into the vessel. The flow should cease when the water has just begun to descend in the lower tube ; if not, the height of the lower shelf must be adjusted until this is the case.

The space above the upper mark is always filled with gas, and that below the lower mark with water, so that the capacity of these portions of the instrument has no effect upon the measurements. The narrow tubes are so small that a variation of even an inch of the level at which the water stands in them has no appreciable effect upon the meter reading.

The apparatus shall only be used in proving a meter when the temperature of the meter and of the water in the water vessel have been found not to differ by more than two degrees Fahrenheit.

In order to prove the meters used in the various testings, the position of the index is taken when the instrument has been put in adjustment and filled with gas as described. The tap of the water vessel is turned off ; the three-way tap is turned half-way towards the position which will connect the instrument with the gas-meter, and the pressure of the gas in the instrument is reduced to atmospheric pressure by momentarily opening the tap in the free branch of the glass T. The water vessel is placed upon the upper shelf, the regulating tap (Fig. 4) is turned on, the three-way tap is turned into such a position as will connect the instrument with the meter, and the tap of the water vessel is turned on. One-twelfth of a cubic foot of gas will then be discharged through the meter. Fig. 20 represents this operation in progress. The three-way stop-cock is then turned so as to fill the instrument with gas, the water vessel is placed upon the lower shelf, the gas is reduced to atmospheric pressure as before, and a second, and again a third quantity is discharged through the meter. Should the hand attached to the

axle of the measuring drum have travelled in the three revolutions as much as one division beyond the point from which it started, some water must be removed from the meter ; if the travel of the meter hand is as much as one division short of this point, some water must be poured in. The operation is then to be repeated until the error is found to fall within the specified limits.

## APPENDIX O.

"Each Gas Examiner shall on each day make and deliver a report of the result of the testings of the gas supplied by the Company conducted by him on the immediately preceding day to the controlling authority, to the Gas Referees, to the Chief Gas Examiner, and to the Company, and the books kept by a Gas Examiner for recording the results of the testing of such gas by him shall be open at all reasonable times to the inspection of the Company without payment." (Gas Light and Coke and other Gas Companies Acts Amendment Act, 1880, Section 11.)

"Each Gas Examiner shall forthwith deliver to the controlling authority, to the Gas Referees, to the Chief Gas Examiner, and to the Company a report of the result of each testing conducted by him under the provisions of this section." (London Gas Act, 1905, Section 5 (2).)

In making his returns for Illuminating Power as given by the flat flame, and for Calorific Power, the Gas Examiner shall put down the result of each testing, and the time at which the testing was made.

On p. 286 is an example of the form in which returns are to be made.



Table giving the Illuminating Power of Gas from Observations of the Rate of Consumption required to yield the Light of 16 Candles, and from Readings of the Barometer, Thermometer, and Refractive Tables, or of the Aerothermometer.

\*The observed time of passage through the meter of one-sixth of a cubic foot of gas is to be found in the left-hand column, and the Aerothermometer Reading or Tabular Number in the top lines. In the corresponding line and column is found the Illuminating Power

Aerothermometer reading Tabular Number	980	940	900	860	820	780	740	700	660	620	580	540	500	460	420	380	340	300	260	220	180	140	100	90	80	70	60	50	40	30	20	10	0		
Minutes. Seconds	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35
1	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
2	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
3	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
4	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
5	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
6	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
7	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
8	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
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10	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
11	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45
12	12-00	13-03	13-17	13-31	13-45	13-59	14-13	14-27	14-41	14-55	15-09	15-23	15-37	15-51	16-05	16-19	16-33	16-47	17-01	17-15	17-29	17-43	17-57	18-11	18-25	18-39	18-53	19-07	19-21	19-35	19-49	20-03	20-17	20-31	20-45

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Time in Seconds.

75 X Tabular Number.

75

Illuminating power =

Aerothermometer Reading X time in Seconds or =

*County of London Gas-Testing Place, 1 Carlyle Square, Chelsea, S. W.*

REPORT ON GAS SUPPLIED BY THE GAS LIGHT AND COKE COMPANY.

DATE :—			
Illuminating Power, in Candles, corrected.			Calorific Power; Calories per Cubic Foot.
Time.	Argand.	Time.	
		Flat Flame.	
			Sulphur in 100 cubic feet of Gas, in grains.  Time..... Gross..... Net .....
			Pressure in tenths of an inch of water.
Mean		Mean	Street..... Time..... Pressure .....

Gas Examiner.

## APPENDIX P.

## LOAN APPARATUS.

In order to make the suspension of any of the operations of gas testing, which occur from time to time owing to a defect in some piece of apparatus, as short as possible, the Gas Referees keep at their office one or two of each of the pieces of apparatus likely to need replacement.

On receiving an application from any Gas Examiner, or from the Chemist to the London County Council, or from the City Gas Examiner, or from the Gas Company, for the temporary loan of a piece of apparatus to take the place of similar apparatus at one of the testing places which needs to be sent away for repair or replaced by new apparatus, the Gas Referees will state whether they have such apparatus in stock, and if so when they will be able to deliver it to the messenger sent to convey it to the testing place.

## APPENDIX B.

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### CYANOGEN IN PURIFYING MATERIALS AND THE INFLUENCE OF AMMONIA UPON ITS FORMATION IN PURIFICATION.

By Dr BURSHELL, Karlsruhe (*Journ. des Usines à Gaz*, 1893).

THE formation of Prussian blue during gas purification is an important matter; researches by Leybold show that it is formed during revivification of the material, the cyanides of iron formed in the purifiers being transformed by oxidation into Prussian blue. It is uncertain whether a cyanide of iron is formed, but it may be assumed that Prussian blue is not formed in the first purifiers because it cannot exist in presence of a large excess of sulphuretted hydrogen.

The absorption of cyanogen in the purifiers is also not due to a double decomposition between the hydrocyanic acid or the cyanide of ammonium and the sulphate of iron, because purifying material which has not taken up sulphuretted hydrogen does not take up cyanogen, or only in a very slight degree. Old material, on the contrary, readily absorbs cyanogen, but it frequently contains sulphocyanogen in the presence of ammonia, which renders it almost useless.

Experiments made by the author prove that cyanogen is taken up in the presence, as well as in the absence, of ammonia.

Ferrocyanogen may be converted into sulphocyanogen, or the reverse, but while ferrocyanogen is easily convertible it is very difficult to effect the contrary conversion.

The presence of ammonia during the purification of gas influences the formation of cyanogen in two ways, and although it may be difficult to avoid ammonia in the purifiers, the formation of sulphocyanogen during revivification may be minimised by avoiding excessive heating, and by removing the ammonia as quickly

as possible by spreading it out in thin layers, &c., when removed from purifiers.

The transformation of cyanogen into sulphocyanogen takes place readily in the presence of ammonia and sulphuretted hydrogen. Sulphocyanogen compounds are formed as well as other cyanogen compounds.

A small portion is arrested in the first purifiers, where the ammonia and sulphuretted hydrogen are taken up in large quantities; the cyanogen then attacks other portions of the purifying material in which there is no sulphuretted hydrogen and only traces of ammonia, which, on account of the oxide of iron present, favours the formation of ferrocyanides, and these portions of the cyanogen may, by a suitable treatment of the purifying material, be utilised for the production of Prussian blue.

#### THE CHEMICAL COMPOSITION AND TECHNICAL ANALYSIS OF WATER-GAS.

By EDWARD H. EARNSHAW (*The American Gas Light Journal*, lxi., 1898).

The oils used in producing carburetted water-gas are decomposed in the fixing chamber with production of hydrogen, gaseous hydrocarbons, naphthalène, benzene, and its homologues.

The constituents of coal-gas and carburetted water-gas are the same, but their proportions vary. The following Table shows the representative analyses:—

Constituents.	Coal-Gas.		Carburetted Water-Gas.	
	Per Cent.		Per Cent.	
Benzene vapour	-	0.50	-	0.60
*Heavy hydrocarbons	-	4.25	-	12.80
Carbonic oxide	-	8.04	-	30.70
Hydrogen	-	47.04	-	32.40
Marsh gas	-	36.02	-	13.90
Higher paraffins	-	0.00	-	2.40
Carbonic acid	-	1.60	-	2.70
Oxygen	-	0.39	-	0.70
Nitrogen	-	2.16	-	3.80
		<hr/> 100.00		<hr/> 100.00

\* The heavy hydrocarbons consist chiefly of ethylene and its homologues.

## ESTIMATION OF FERROCYANIDE IN SPENT OXIDE.

By E. DONATH and B. MOYOSCHES (*Zeits. für Angew. Chem.*, 1899, pp. 345-347).

50 grams of the powdered material (or the residue after the sulphur has been extracted) are taken, placed in a litre flask, and 100 to 150 c.c. of 15 per cent. solution of caustic potash added, and heated on a sand bath, with vigorous shakings, for some time. The volume is then made up to 1,030 c.c. with distilled water (the residue from 50 grams occupies a volume equal to 30 c.c., and the liquid is equal to 1,000 c.c. net). It is then filtered off, and an aliquot part of the filtrate utilised for estimation. 80 grams of sodium hydroxide are dissolved in water and made up to 1 litre and thoroughly well agitated, 20 c.c. bromine then added, and well mixed. This oxidising agent can be added to filtrate to be tested as required. The mixture is then boiled until gas is given off, and a brick-red precipitate is thrown down. The precipitate is filtered off and afterwards dissolved in hydrochloric acid, and the proportion of iron determined by permanganate test. The amount of iron  $\times 7.5476$  gives the equivalent of potassium ferrocyanide,  $K_4FeCy_6$ , and this multiplied by .687 gives the Prussian blue.

## PRUSSIAN BLUE IN SPENT OXIDE BY FELD'S METHOD.

R. WITZECK (*Journ. Soc. Chem. Ind.*, vol. xxiii., p. 13, extracted from *Journ. für Gasbeleuchtung*).

The total cyanogen is determined by pulverising in a mortar 2 grams of spent oxide (0.5 gram of mud) with 1 c.c. of  $\frac{N}{1}$  ferrous sulphate solution, and 5 c.c. of 8N sodium hydrate solution for five minutes; 30 c.c. of 3N magnesium chloride solution are added, drop by drop, with continuous stirring, the contents being washed into a distillation flask of about 700 c.c. capacity, and the washing, &c., made up to about 200 c.c. After a few minutes' boiling, 100 c.c. of boiling  $\frac{N}{10}$  mercuric chloride solution are added to the boiling liquid, and the mixture boiled for ten minutes, all the cyanogen being thus converted into mercuric cyanide.

The flask is now attached to a condenser leading into a receiver, the latter being connected to a safety-bulb tube; 30 c.c. of 4N sulphuric acid are added and the liquid is distilled for about thirty minutes, the products passing over into the receiver and bulb, which contain 20 c.c. of 2N sodium hydrate. Sometimes the distillate is turbid owing to presence of sulphur, if so, an excess of lead carbonate is added, agitate and filter, and an aliquot portion taken for titration. This is performed by adding 5 c.c. of 4N potassium iodide solution, and then titrate with  $\frac{N}{10}$  silver nitrate solution until a yellow turbidity appears.

1 c.c. of  $\frac{N}{10}$  silver nitrate = .009556 gram of Prussian blue.

#### MONAZITE SANDS.

Dr C. RICHARD BÖHM (*Journal of Gas Lighting*, vol. xciii, p. 430).

The average analysis of four samples by Hussak and Reitniger of a monazite from Bandeira de Mello are given under I., and of a sample from Bandeirenha in Minas-Geraes, II.

	I.	II.
	Per cent.	Per cent.
P <sub>2</sub> O <sub>5</sub>	25.51	29.18
Ce <sub>2</sub> O <sub>3</sub>	32.14	32.46
Nd <sub>2</sub> O <sub>3</sub>	15.38	16.81
(LaFr) <sub>2</sub> O <sub>3</sub>	10.61	19.21
ThO <sub>2</sub>	10.05	1.09
Fe <sub>2</sub> O <sub>3</sub>	1.79	0.61
CaO	0.20	0.10
ZrO <sub>2</sub>	0.60	...
Al <sub>2</sub> O <sub>3</sub>	0.84	...
SiO <sub>2</sub>	2.63	...
H <sub>2</sub> O	0.92	...
	100.67	99.46

#### HUMIDITY, EFFECT OF, ON THE PENTANE LAMP.

(*Journal of Gas Lighting*, vol. xciii., p. 162.)

This subject was dealt with in the Report of the Research Committee of the American Gas-Light Association, by Mr J. B. Klump.

The object was to present a means of comparing photometric standards or other lights represented by open flame burners, with vacuum electric standards or such lights as are not affected by varying atmospheric conditions. Attention was drawn to the work on the same subject by Dr Liebenthal of Berlin in 1895, and by C. C. Paterson of the National Physical Laboratory in 1904.

These show that the standard light is affected by the presence of carbonic acid, barometric pressure, and moisture. For commercial purposes the variation due to carbonic acid may be omitted.

With regard to barometric pressure Dr Liebenthal has shown that 25 mm. of mercury affected the Hefner lamp 0.28 per cent., and the 1 candle pentane lamp 1.2 per cent.

Mr Paterson's readings under a maximum variation of the barometer from 739 to 780 mm. show a variation of lamp value of less than 1.6 per cent. from the normal. The effect of moisture is shown to be considerable. A variation of 1 per cent. in candle-power for each 1.515 litre of water vapour per cubic metre of dry air is obtained. He states that with a temperature of 70 degrees Fahr. and a humidity of 60 per cent., the variation will be 3.3 per cent., *i.e.*, the actual light-giving value of the 10-candle pentane lamp will be 9.67 candles.

To show the necessity of making corrections, the author gives a Table of the average humidity for Philadelphia for the past thirty-four years, from which it is seen that the moisture varies from 4.2 to 20.2 litres per cubic metre, and the percentage variation in candle-power ranges from +3.4 to -7.2.

### ESTIMATION OF CARBON BISULPHIDE IN BENZENE.

By D. STAROVINUS (*Journ. Gasbeleuchtung*, p. 8).

25 c.c. of the sample are mixed with 70 c.c. of 96 per cent. alcohol, and 10 c.c. 2N sodium hydroxide. After half an hour 5 c.c. of concentrated hydrogen peroxide are added, the alcohol evaporated off, and the sulphate formed precipitated by barium chloride, and estimated as usual.

The method can also be used volumetrically by using 10 c.c. of normal alkali, and titrating the excess with  $\frac{N}{5}$  acid, using methyl orange as indicator.

1 c.c. of  $\frac{N}{5}$  alkali = 0.019 gram of carbon bisulphide.



ESTIMATION OF CS<sub>2</sub> AND S IN COMMERCIAL BENZENE.

By ED. S. JOHNSON (*Journ. Amer. Chem. Soc.*, p. 1209).

**CS<sub>2</sub>.**—This is converted into potass xanthate by saturated alcoholic potassium hydrate, and is removed from the benzene by repeated washings with alkaline water. The xanthate is then converted into the copper compound by slightly acidifying with acetic acid, and precipitating with copper sulphate. The precipitate is filtered, washed, and ignited to oxide.

One part of oxide = 1.75 of carbon disulphide.

**Total Sulphur.**—5 c.c. of the sample are vaporised by a current of hydrogen, they are then burnt in a special apparatus in oxygen, and the products of combustion are absorbed in bottles containing sodium carbonate and bromine solution. This causes a complete conversion of the sulphur into sulphuric acid, which may either be estimated gravimetrically or volumetrically by collecting the products of combustion in a standard solution of sodium hydrate, and, after adding some neutral hydrogen peroxide, the excess of alkali is titrated back with standard acid.

## TEST FOR CN IN PRESENCE OF HCN.

By THEODOR WALLIS (*Journal Chem. Soc.*, from "Annalen," pp. 353-362).

The method for the detection of cyanogen in the presence of hydrogen cyanide:—The mixture is passed into an acidified solution of a silver salt, the silver cyanide removed, the silver in the solution precipitated by yellow ammonium sulphide, a few drops of alkali are added, and after filtration and evaporation the test for thiocyanate is applied. The estimation of cyanogen and hydrogen cyanide is effected by passing the mixture into a solution of sodium hydroxide, the HCN and one-half of the CN is estimated as silver cyanide; the other half, now present as potassium cyanate, being determined by boiling the cyanate with dilute sulphuric acid and titrating the ammonia produced.

An alternative method is to pass the mixture into a solution of ammonium hydroxide or carbonate. After titration with a silver solution, the ammonium cyanate is converted into carbamide, and isolated and weighed as such.

Cyanogen prepared in the usual way always contains HCN, which can be removed by passing the gas over cotton wool moistened with a silver solution.

## ESTIMATION OF BENZOL IN GAS.

Messrs. Hoffmann and Küspert found that benzol, with an ammoniacal solution of cyanide of nickel, forms a bluish-white precipitate, consisting of an equal number of molecules of cyanide of nickel, ammonia, and benzol. The benzol is held in so firm a combination that it cannot be displaced either by cold water or by ammonia, though it can be taken out by large quantities of ether. On boiling with water or cyanide solution the benzol is set free. In the absence of ammonia no such precipitate is formed; and an ammoniacal solution of nickel hydrate gives no results. Dennis and O'Neill, unwilling to work with poisonous cyanide of nickel, found that an ammoniacal solution of nitrate of nickel works equally well; and their solution is made of 14 grams of nitrate of nickel, nitric acid (sp. gr. 1.44) 2 c.c., water 160 c.c., to form a solution, which is then slowly poured into 100 c.c. ammonia of sp. gr. 0.908. The solution is now ready for use, but it is better to let it stand for some hours to allow a little double salt to crystallise out.

The gas is shaken up with the solution in a Hempel pipette, but as it picks up ammonia from the solution, and increases a little in bulk, it has therefore to be then shaken up with 10 per cent sulphuric acid to remove this ammonia. The final volume shows by difference the volume of benzol absorbed.

## APPENDIX C.

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### WEIGHTS AND MEASURES.

THE Weights and Measures Act of 1897 made the use of the metric system permissible in the trade of the United Kingdom, but unfortunately, owing to various circumstances, this permission has not been taken advantage of.

The Board of Trade standards are derived from the standard metre and the standard kilogramme, and the imperial equivalents of these are given by Order of Council dated 19th May 1898, and are as follows :—

1 metre	= 39.370113 inches.
1 „	= 3.280843 feet.
1 „	= 1.0936143 yards.
1 kilogramme	= 2.2046223 lbs.
1 „	= 15432.35 grains.

From the metre all other measures and weights are derived.

Deka, hecto, kilo mean respectively ten, a hundred, a thousand; and deci, centi, milli mean respectively a tenth, a hundredth, a thousandth.

#### *Liquid—*

1 cubic centimetre = 1 gram of water at 4° Cent.

1 kilogramme = the weight of 1 litre of water at 4° Cent.

*Liquid*

1 litre	100 centilitres = 10 decilitres = $\frac{1}{10}$ dekalitre = $\frac{1}{100}$ hectolitre = 1000.16 cubic centimetres; or in English measure,
1 litre	1.75980 pints or 0.22 gallon.
1 gallon	4.5459631 litres.
1 gram	15.432 grains.
1,000 kilogrammes	0.9182 ton.
16 oz.	1 lb. 7,000 grains = 0.45359243 kilogramme.
0.00648 gram	1 grain.
1 lb. (Avoirdupois)	453.593 grams.

*Cubic Measure*

1,728 cubic inches	= 1 cubic foot.
27 " feet	= 1 " yard.

*Useful Memoranda.*

Weight of a cubic foot of hydrogen	37.15 grains.
" " " carbonic acid	817.30 "
" " " sulphuretted hydrogen	631.54 "
" " " ammonia	315.77 "
" " " carbonic oxide	520.10 "
" " " marsh gas	297.20 "
" " " nitrogen	520.10 "
" " " olefiant gas	520.10 "
" " " air	535.96 "
" " " oxygen	594.40 "
" " " water vapour	334.35 "
" " " bisulphide of carbon	1411.70 "

In order to obtain the weight of a cubic foot of any gas whose molecular formula is known, calculate the molecular weight; divide this by 2, and multiply by the weight of 1 cubic foot of hydrogen (37.15). This will give the weight in grains of 1 cubic foot of the gas.

*Example.*

The molecular weight of carbonic acid =  $44 \div 2 = 22$ .  
 $22 \times 37.15 = 817.30$  grains per cubic foot.

## COMPARISON OF THERMOMETERS.

Centi- grade.	Fahren- heit.	Centi- grade.	Fahren- heit.	Centi- grade.	Fahren- heit.	Centi- grade.	Fahren- heit.
+ 260	+ 500.0	+ 225	+ 437.0	+ 190	+ 374.0	+ 155	+ 311.0
259	498.2	224	435.2	189	372.2	154	309.2
258	496.4	223	433.4	188	370.4	153	307.4
257	494.6	222	431.6	187	368.6	152	305.6
256	492.8	221	429.8	186	366.8	151	303.8
255	491.0	220	428.0	185	365.0	150	302.0
254	489.2	219	426.2	184	363.2	149	300.2
253	487.4	218	424.4	183	361.4	148	298.4
252	485.6	217	422.6	182	359.6	147	296.6
251	483.8	216	420.8	181	357.8	146	294.8
250	482.0	215	419.0	180	356.0	145	293.0
249	480.2	214	417.2	179	354.2	144	291.2
248	478.4	213	415.4	178	352.4	143	289.4
247	476.6	212	413.6	177	350.6	142	287.6
246	474.8	211	411.8	176	348.8	141	285.8
245	473.0	210	410.0	175	347.0	140	284.0
244	471.2	209	408.2	174	345.2	139	282.2
243	469.4	208	406.4	173	343.4	138	280.4
242	467.6	207	404.6	172	341.6	137	278.6
241	465.8	206	402.8	171	339.8	136	276.8
240	464.0	205	401.0	170	338.0	135	275.0
239	462.2	204	399.2	169	336.2	134	273.2
238	460.4	203	397.4	168	334.4	133	271.4
237	458.6	202	395.6	167	332.6	132	269.6
236	456.8	201	393.8	166	330.8	131	267.8
235	455.0	200	392.0	165	329.0	130	266.0
234	453.2	199	390.2	164	327.2	129	264.2
233	451.4	198	388.4	163	325.4	128	262.4
232	449.6	197	386.6	162	323.6	127	260.6
231	447.8	196	384.8	161	321.8	126	258.8
230	446.0	195	383.0	160	320.0	125	257.0
229	444.2	194	381.2	159	318.2	124	255.2
228	442.4	193	379.4	158	316.4	123	253.4
227	440.6	192	377.6	157	314.6	122	251.6
226	438.8	191	375.8	156	312.8	121	249.8

COMPARISON OF THERMOMETERS—*Continued.*

Centi- grade.	Fahren- heit.	Centi- grade.	Fahren- heit.	Centi- grade.	Fahren- heit.	Centi- grade.	Fahren- heit.
+ 120	+ 248.0	+ 85	+ 185.0	+ 50	+ 122.0	+ 15	59.0
119	246.2	84	183.2	49	120.2	14	57.2
118	244.4	83	181.4	48	118.4	13	55.4
117	242.6	82	179.6	47	116.6	12	53.6
116	240.8	81	177.8	46	114.8	11	51.8
115	239.0	80	176.0	45	113.0	10	50.0
114	237.2	79	174.2	44	111.2	9	48.2
113	235.4	78	172.4	43	109.4	8	46.4
112	233.6	77	170.6	42	107.6	7	44.6
111	231.8	76	168.8	41	105.8	6	42.8
110	230.0	75	167.0	40	104.0	5	41.0
109	228.2	74	165.2	39	102.2	4	39.2
108	226.4	73	163.4	38	100.4	3	37.4
107	224.6	72	161.6	37	98.6	2	35.6
106	222.8	71	159.8	36	96.8	1	33.8
						Water freezes	
105	221.0	70	158.0	35	95.0	0	32.0
104	219.2	69	156.2	34	93.2	— 1	30.2
103	217.4	68	154.4	33	91.4	2	28.4
102	215.6	67	152.6	32	89.6	3	26.6
101	213.8	66	150.8	31	87.8	4	24.8
Water boils							
100	212.0	65	149.0	30	86.0	5	23.0
99	210.2	64	147.2	29	84.2	6	21.2
98	208.4	63	145.4	28	82.4	7	19.4
97	206.6	62	143.6	27	80.6	8	17.6
96	204.8	61	141.8	26	78.8	9	15.8
						10	14.0
95	203.0	60	140.0	25	77.0	...	...
94	201.2	59	138.2	24	75.2	...	...
93	199.4	58	136.4	23	73.4	...	...
92	197.6	57	134.6	22	71.6	...	...
91	195.8	56	132.8	21	69.8	...	...
90	194.0	55	131.0	20	68.0	...	...
89	192.2	54	129.2	19	66.2	...	...
88	190.4	53	127.4	18	64.4	...	...
87	188.6	52	125.6	17	62.6	...	...
86	186.8	51	123.8	16	60.8	...	...

## CONVERSION OF THERMOMETER DEGREES.

° 9, divide by 5, then add 32.  
then multiply by 5, and divide by 9.

## COMPARISON OF DIFFERENT HYDROMETERS. 299

## COMPARISON OF DIFFERENT HYDROMETERS.

(Degrees according to Baumé and Twaddell, with the  
Specific Gravities.)

B.	T.	Specific Gravity.	B.	T.	Specific Gravity.	B.	T.	Specific Gravity.
0.0	0.0	1.000	17.7	28.0	1.140	32.8	59.0	1.295
0.7	1.0	1.005	18.0	28.4	1.142	33.0	59.4	1.297
1.0	1.4	1.007	18.3	29.0	1.145	33.3	60.0	1.300
1.4	2.0	1.010	18.8	30.0	1.150	33.7	61.0	1.305
2.0	2.8	1.014	19.0	30.4	1.152	34.0	61.6	1.308
2.1	3.0	1.015	19.3	31.0	1.155	34.2	62.0	1.310
2.7	4.0	1.020	19.8	32.0	1.160	34.6	63.0	1.315
3.0	4.4	1.022	20.0	32.4	1.162	35.0	64.0	1.320
3.4	5.0	1.025	20.3	33.0	1.165	35.4	65.0	1.325
4.0	5.8	1.029	20.9	34.0	1.170	35.8	66.0	1.330
4.1	6.0	1.030	21.0	34.2	1.171	36.0	66.4	1.332
4.7	7.0	1.035	21.4	35.0	1.175	36.2	67.0	1.335
5.0	7.4	1.037	22.0	36.0	1.180	36.6	68.0	1.340
5.4	8.0	1.040	22.5	37.0	1.185	37.0	69.0	1.345
6.0	9.0	1.045	23.0	38.0	1.190	37.4	70.0	1.350
6.7	10.0	1.050	23.5	39.0	1.195	37.8	71.0	1.355
7.0	10.2	1.052	24.0	40.0	1.200	38.0	71.4	1.357
7.4	11.0	1.055	24.5	41.0	1.205	38.2	72.0	1.360
8.0	12.0	1.060	25.0	42.0	1.210	38.6	73.0	1.365
8.7	13.0	1.065	25.5	43.0	1.215	39.0	74.0	1.370
9.0	13.4	1.067	26.0	44.0	1.220	39.4	75.0	1.375
9.4	14.0	1.070	26.4	45.0	1.225	39.8	76.0	1.380
10.0	15.0	1.075	26.9	46.0	1.230	40.0	76.6	1.383
10.6	16.0	1.080	27.0	46.2	1.231	40.1	77.0	1.385
11.0	16.6	1.083	27.4	47.0	1.235	40.5	78.0	1.390
11.2	17.0	1.085	27.9	48.0	1.240	40.8	79.0	1.395
11.9	18.0	1.090	28.0	48.2	1.241	41.0	79.4	1.397
12.0	18.2	1.091	28.4	49.0	1.245	41.2	80.0	1.400
12.4	19.0	1.095	28.8	50.0	1.250	41.6	81.0	1.405
13.0	20.0	1.100	29.0	50.4	1.252	42.0	82.0	1.410
13.6	21.0	1.105	29.3	51.0	1.255	42.3	83.0	1.415
14.0	21.6	1.108	29.7	52.0	1.260	42.7	84.0	1.420
14.2	22.0	1.110	30.0	52.6	1.263	43.0	84.8	1.424
14.9	23.0	1.115	30.2	53.0	1.265	43.1	85.0	1.425
15.0	23.2	1.116	30.6	54.0	1.270	43.4	86.0	1.430
15.4	24.0	1.120	31.0	54.8	1.274	43.8	87.0	1.435
16.0	25.0	1.125	31.1	55.0	1.275	44.0	87.6	1.438
16.5	26.0	1.130	31.5	56.0	1.280	44.1	88.0	1.440
17.0	26.8	1.134	32.0	57.0	1.285	44.4	89.0	1.445
17.1	27.0	1.135	32.4	58.0	1.290	44.8	90.0	1.450

COMPARISON OF DIFFERENT HYDROMETERS—*Continued.*

B.	T.	Specific Gravity.	B.	T.	Specific Gravity.	B.	T.	Specific Gravity.
45.0	90.6	1.453	54.0	119.4	1.597	61.8	150.0	1.750
45.1	91.0	1.455	54.1	120.0	1.600	62.0	150.6	1.753
45.4	92.0	1.460	54.4	121.0	1.605	62.1	151.0	1.755
45.8	93.0	1.465	54.7	122.0	1.610	62.3	152.0	1.760
46.0	93.6	1.468	55.0	123.0	1.615	62.5	153.0	1.765
46.1	94.0	1.470	55.2	124.0	1.620	62.8	154.0	1.770
46.4	95.0	1.475	55.5	125.0	1.625	63.0	155.0	1.775
46.8	96.0	1.480	55.8	126.0	1.630	63.2	156.0	1.780
47.0	96.6	1.483	56.0	127.0	1.635	63.5	157.0	1.785
47.1	97.0	1.485	56.3	128.0	1.640	63.7	158.0	1.790
47.4	98.0	1.490	56.6	129.0	1.645	64.0	159.0	1.795
47.8	99.0	1.495	56.9	130.0	1.650	64.2	160.0	1.800
48.0	99.6	1.498	57.0	130.4	1.652	64.4	161.0	1.805
48.1	100.0	1.500	57.1	131.0	1.655	64.6	162.0	1.810
48.4	101.0	1.505	57.4	132.0	1.660	64.8	163.0	1.815
48.7	102.0	1.510	57.7	133.0	1.665	65.0	164.0	1.820
49.0	103.0	1.515	57.9	134.0	1.670	65.2	165.0	1.825
49.4	104.0	1.520	58.0	134.2	1.671	65.5	166.0	1.830
49.7	105.0	1.525	58.2	135.0	1.675	65.7	167.0	1.835
50.0	106.0	1.530	58.4	136.0	1.680	65.9	168.0	1.840
50.3	107.0	1.535	58.7	137.0	1.685	66.0	168.4	1.842
50.6	108.0	1.540	58.9	138.0	1.690	66.1	169.0	1.845
50.9	109.0	1.545	59.0	138.2	1.691	66.3	170.0	1.850
51.0	109.2	1.546	59.2	139.0	1.695	66.5	171.0	1.855
51.2	110.0	1.550	59.5	140.0	1.700	66.7	172.0	1.860
51.5	111.0	1.555	59.7	141.0	1.705	67.0	173.0	1.865
51.8	112.0	1.560	60.0	142.0	1.710	...	...	...
52.0	112.6	1.563	60.2	143.0	1.715	...	...	...
52.1	113.0	1.565	60.4	144.0	1.720	...	...	...
52.4	114.0	1.570	60.6	145.0	1.725	...	...	...
52.7	115.0	1.575	60.9	146.0	1.730	...	...	...
53.0	116.0	1.580	61.0	146.4	1.732	...	...	...
53.3	117.0	1.585	61.1	147.0	1.735	...	...	...
53.6	118.0	1.590	61.4	148.0	1.740	...	...	...
53.9	119.0	1.595	61.6	149.0	1.745	...	...	...

*N. B.*—The Baumé degrees are calculated by the formula—

$$d = \frac{144.3}{144.3 - N} \text{ water of } 15^{\circ} \text{ Cent.}$$

being put = 0°, and sulphuric acid of 1.842 at 15° Cent. = 66°.

Compare Lunge's "Sulphuric Acid and Alkali," vol. i., p. 20.

This is the Baumé's hydrometer, mostly used on the Continent of Europe, but other scales are in use there as well, and quite another scale for Baumé's hydrometer is used in America.



# SPECIFIC GRAVITY, SPECIFIC HEAT OF GASES. 301

## SPECIFIC GRAVITY AND WEIGHTS OF GASES.

Description.	Specific Gravity (air=1,000)	Weight per 100 cub. ft.	Description.	Specific Gravity (air=1,000)	Weight per 100 cub. ft.
Air	1.000	7.66	Coal-gas	0.500	3.83
Aqueous vapour	0.633	4.85	Hydrochloric acid	1.247	9.55
Ammonia	0.589	4.51	Hydrogen	0.069	0.53
Carbonic acid	1.529	11.71	Marsh gas	0.562	4.30
oxide	0.967	7.41	Nitric oxide	1.039	7.95
Carbonuretted hydro-			Nitrous oxide	1.527	11.70
gen, heavy	0.978	7.49	Nitrogen	0.971	7.44
Carbonuretted hydro-			Olefiant gas	0.982	7.52
gen, light	0.557	4.26	Oxygen	1.105	8.46
Chlorine	2.470	18.92	Steam, gaseous	0.622	4.76

## SPECIFIC HEAT.

The Specific Heat of a body is the ratio of the quantity of heat required to raise that body 1° to the quantity required to raise an equal weight of water 1°. Example:—Water is the standard=1. The specific heat of mercury is .033. Therefore the quantity of heat required to heat 1 lb. of mercury 1° Fahr. would heat 1 lb. of water .033° Fahr.; or the quantity of heat required to heat 1 lb. of water 1° Fahr. would heat 30 lb. of mercury 1° Fahr.

### TABLE OF SPECIFIC HEATS.

Water	-	-	1.000	Carbonic acid	-	0.216 and 0.171
Acetic acid	-	-	0.659	oxide	-	0.248 and 0.177
Acetone vapour	-	-	0.413	Chalk	-	0.215
Air	-	*0.238 and	0.169	Charcoal, wood	-	0.241
Alcohol	-	-	0.659	animal	-	0.261
vapour	-	-	0.453	Chlorine gas	-	0.121
Aluminium	-	-	0.234	Clay	-	0.185
Ammonia gas	-	-	0.508	Coal (anthracite)	-	0.201
liquor (sp. gr.	-	-		(bituminous)	-	0.241
1.024)	-	-	0.887	Coke (increases as tem-	-	
Beech wax	-	-	0.45	perature rises)	-	0.203
Benzene	-	-	0.394	Copper	-	0.095
Benzol	-	-	0.381	Creosote	-	0.458
vapour	-	-	0.375	Ether	-	0.521
Dioxide of nitrogen	-	-	0.231	vapour	-	0.481
Hickwood	-	-	0.48	Ethylene gas	-	0.404
Disulphide carbon vapour	-	-	0.157	Glass	-	0.194
Brass	-	-	0.094	Gold	-	0.032
Brick work	-	-	0.192	Graphite	-	0.202
fire	-	-	0.22	Hydrochloric acid (sol.)	-	0.600
Bromine vapour	-	-	0.056	"    (gas)	-	0.185
Carbon	-	-	0.241	Hydrogen	-	3.405 and 2.410

\* Where two specific heats are given for a gas, these are the specific heats for the gas at constant pressure and at constant volume respectively. It takes .238 B.T.U. to heat a pound of air 1° Fahr. when it is allowed to expand or to keep the pressure constant, and only .169 B.T.U. to do the same when the gas is prevented from doing so. The reason is that no heat is, in the latter case, expended in doing external work.

TABLE OF SPECIFIC HEATS—*Continued.*

Ice -	-	-	0.504	Spermaceti -	-	0.32
Iron, cast -	-	-	0.130	Steam (saturated at constant volume) -	-	0.305
„ wrought -	-	-	0.110	„ (gas) -	0.475 and	0.370
Lead -	-	-	0.031	Steel -	-	0.117
Lime, burned -	-	-	0.217	Stonework -	-	0.197
Magnesium -	-	-	0.250	Sulphur -	-	0.203
Marble, white -	-	-	0.216	Sulphuretted hydrogen -	-	0.243
Marsh gas -	-	-	0.593	Sulphuric acid, density 1.87	-	0.335
Mercury -	-	-	0.033	„ „ „ 1.30	-	0.661
Nitric oxide -	-	-	0.232	Sulphurous anhydride -	-	0.155
Nitrogen -	0.244 and	0.174	-	Tar, sp. gr. 1.176 -	-	0.288
Nitrous oxide -	-	-	0.226	Tin -	-	0.056
Oil, olive -	-	-	0.310	Turpentine oil -	-	0.416
Oxygen gas -	0.218 and	0.156	-	„ vapour -	-	0.506
Petroleum -	-	-	0.434	Water at 32° Fahr. -	-	1.000
Phosphorus -	-	-	0.250	„ 212° Fahr. -	-	1.013
Pine -	-	-	0.65	Wood (average) -	-	0.550
Platinum -	-	-	0.034	„ spirit -	-	0.601
Potassium -	-	-	0.167	Zinc -	-	0.093
Silver -	-	-	0.056			

SPECIFIC GRAVITY AND WEIGHTS OF VARIOUS LIQUIDS.

VARIOUS.				OILS.			
Description.	Specific Gravity (Water = 1).	Weight in lbs.		Description.	Specific Gravity (Water = 1,000).	Weight in lbs.	
		Cubic feet.	Per gallon.			Cubic feet.	Per gallon.
Acid—				Almond oil (59° Fahr.) -	0.918	57	9.18
Hydrochloric -	1.27	75	12.7	Castor oil -	0.97	60	9.7
Nitric -	1.22	75	12.2	Cod liver oil -	0.92	57	9.2
Sulphuric -	1.84	115	18.4	Essential oil of bitter almonds	1.049	65	10.49
Alcohol—				Essential oil of turpentine -	0.864	54	8.64
Absolute -	0.80	50	8.0	Fat, animal -	0.92	57	9.2
Proof -	0.92	57	9.2	Lard oil -	0.91	57	9.1
Ammonium, liquid -	0.73	45	7.3	Linseed oil (53° Fahr.) -	0.94	59	9.4
Benzene -	0.85	53	8.5	Mineral lubricating oil -	0.89	55	8.9
Benzol -	0.85	53	8.5	Neatsfoot oil -	0.91	57	9.1
Mercury (at 32° Fahr.) -	13.59	847	135.9	Olive oil -	0.92	57	9.2
Milk -	1.03	64	10.3	Paraffin oil -	0.82	51	8.2
Naphtha, coal -	0.85	53	8.5	„ spirit -	0.78	49	7.8
„ wood -	0.80	50	8.0	Rape and colza oil -	0.91	57	9.1
Petroleum -	0.88	55	8.8	Tallow -	0.94	59	9.4
Water—				Train oil -	0.93	58	9.3
Distilled -	1.000	62	10.0	Turpentine -	0.87	54	8.7
Rain -	1.001	62	10.01				
Sea -	1.026	64	10.26				
Fine, say -	1.014	63	10.14				

## DENSITIES AND WEIGHTS OF GASES AND VAPOURS.

*(Winkler and Lunge's "Technical Gas Analysis.")*

Name of Gas.	Molecular Formula.	Density. (Hydrogen = 1.)	1 Litre of Gas in the Normal State weighs
			Grammes.
Acetylene - - -	$C_2H_2$	12.970	1.1621
Benzene - - -	$C_6H_6$	38.910	3.4863
Butylene - - -	$C_4H_8$	27.940	2.5034
Carbon disulphide - - -	$CS_2$	37.965	3.4017
„ oxysulphide - - -	$CO_2S$	29.955	2.6839
Cyanogen - - -	$(CN)_2$	25.990	2.3287
Ethane - - -	$C_2H_6$	14.970	1.2413
Hydrogen cyanide - - -	$HCN$	13.495	1.2091
Phosphoretted hydrogen - - -	$H_3P$	16.980	1.5214
Propylene - - -	$C_3H_6$	20.955	1.8775
Silicium tetrafluoride - - -	$SiF_4$	52.055	4.6641

## SPECIFIC GRAVITIES AND WEIGHTS OF GASES AND VAPOURS.

*(“Alkali Maker's Handbook.”)*North latitude,  $52^\circ 30'$ , 130 feet above sea level.

Gas.	Symbol.	Molecular Weight.	Specific Gravity (Air=1.)	Grains per cub. ft. $29.92''$ and $32''$ Fahr.	Lbs. per cub. foot $29.92''$ and $32''$ Fahr.*
Ammonia - - -	$NH_3$	17.0	0.58890	332.96	.04757
Atmospheric air - - -	...	...	1.00000	565.16	.08074
Bromine - - -	$Br_2$	160.0	5.52271	3122.1	.4460
Chlorine - - -	$Cl_2$	71.0	2.44921	1384.73	.1978
Carbonic oxide - - -	$CO$	28.0	0.96709	546.78	.07811
„ anhydride - - -	$CO_2$	44.0	1.51968	859.21	.12274
Ethylene - - -	$C_2H_4$	28.0	0.96744	546.98	.07814
Hydrogen - - -	$H_2$	2.0	0.06923	39.1439	.0055919
„ chloride - - -	$HCL$	36.5	1.25922	711.94	.1017
Iodine - - -	$I_2$	254.0	8.756	4949.90	.7071
Methane - - -	$CH_4$	16.0	0.55297	312.64	.04466
Mercury - - -	$Hg$	200.0	...	3914.39	.5592
Nitrogen - - -	$N_2$	28.0	0.97010	548.47	.07835
Nitrous oxide - - -	$N_2O$	44.0	1.52269	860.90	.1229
Nitric oxide - - -	$NO$	30.0	1.03767	586.66	.08381
Nitrous anhydride - - -	$N_2O_3$	76.0	2.630	1487.46	.2125
Nitric peroxide - - -	$NO_2$	46.0	1.592	900.31	.1286
„ „ - - -	$N_2O_4$	92.0	3.184	1800.63	.2572
Oxygen - - -	$O_2$	32.0	1.10521	624.85	.08926
Sulphuretted hydrogen - - -	$H_2S$	34.0	1.17697	665.44	.09506
Sulphurous anhydride - - -	$SO_2$	64.0	2.21295	1251.19	.1787
Sulphur - - -	$S_2$	64.0	2.2155	1252.59	.1789
Water - - -	$H_2O$	18.0	0.62182	351.57	.05022

\* For calculations with large quantities of gas, it is sufficiently accurate to assume that 10,000 cub. ft. weigh as many cwt. as the molecular weight of the gas divided by 4 indicates.

For example, 10,000 cub. ft. of sulphuretted hydrogen weigh  $\frac{34}{4} = 8.5$  cwt. (exactly, it would be 8.488 cwt.).

## SPECIFIC GRAVITIES OF LIQUOR AMMONIA AT 15° CENT.

*(Lunge and Wiernik.)*

Specific Gravity at 15°.	Per cent. $\text{NH}_3$ .	1 Litre contains grms. $\text{NH}_3$ .	Correction of the Specific Gravity for $\pm 1^\circ \text{C}$ .	Specific Gravity at 15°.	Per cent. $\text{NH}_3$ .	1 Litre contains grms. $\text{NH}_3$ .	Correction of the Specific Gravity for $\pm 1^\circ \text{C}$ .
1.000	0.00	0.0	0.00018	0.940	15.63	146.9	0.00039
0.998	0.45	4.5	0.00018	0.938	16.22	152.1	0.00040
0.996	0.91	9.1	0.00019	0.936	16.82	157.4	0.00041
0.994	1.37	13.6	0.00019	0.934	17.42	162.7	0.00041
0.992	1.84	18.2	0.00020	0.932	18.03	168.1	0.00042
0.990	2.31	22.9	0.00020	0.930	18.64	173.4	0.00042
0.988	2.80	27.7	0.00021	0.928	19.25	178.6	0.00043
0.986	3.30	32.5	0.00021	0.926	19.87	184.2	0.00044
0.984	3.80	37.4	0.00022	0.924	20.49	189.3	0.00045
0.982	4.30	42.2	0.00022	0.922	21.12	194.7	0.00046
0.980	4.80	47.0	0.00023	0.920	21.75	200.1	0.00047
0.978	5.30	51.8	0.00023	0.918	22.39	205.6	0.00048
0.976	5.80	56.6	0.00024	0.916	23.03	210.9	0.00049
0.974	6.30	61.4	0.00024	0.914	23.68	216.3	0.00050
0.972	6.80	66.1	0.00025	0.912	24.33	221.9	0.00051
0.970	7.31	70.9	0.00025	0.910	24.99	227.4	0.00052
0.968	7.82	75.7	0.00026	0.908	25.65	232.9	0.00053
0.966	8.33	80.5	0.00026	0.906	26.31	238.3	0.00054
0.964	8.84	85.2	0.00027	0.904	26.98	243.9	0.00055
0.962	9.35	89.9	0.00028	0.902	27.65	249.4	0.00056
0.960	9.91	95.1	0.00029	0.900	28.33	255.0	0.00057
0.958	10.47	100.3	0.00030	0.898	29.01	260.5	0.00058
0.956	11.03	105.4	0.00031	0.896	29.69	266.0	0.00059
0.954	11.60	110.7	0.00032	0.894	30.37	271.5	0.00060
0.952	12.17	115.9	0.00033	0.892	31.05	277.0	0.00060
0.950	12.74	121.0	0.00034	0.890	31.75	282.6	0.00061
0.948	13.31	126.2	0.00035	0.888	32.50	288.6	0.00062
0.946	13.88	131.3	0.00036	0.886	33.25	294.6	0.00063
0.944	14.46	136.5	0.00037	0.884	34.10	301.4	0.00064
0.942	15.04	141.7	0.00038	0.882	34.95	308.3	0.00065

# CALORIFIC POWER OF VARIOUS COMBUSTIBLES. 305

## CALORIFIC POWER OF VARIOUS COMBUSTIBLES.

Constituent.	B.T.U.	C.U.
Carbon to carbon dioxide - - -	14,500	8,080
Carbon to carbon monoxide - - -	4,327	2,404
Hydrogen to water - - -	61,524	34,180
Methane to carbon dioxide and water -	24,513	13,063
Ethylene - - -	21,345	11,858
Sulphur to sulphur dioxide - - -	3,891	2,162
Wood - - -	4,500 to 7,200	2,500 to 4,000
Coal - - -	11,500 to 16,200	6,500 to 9,000
Natural oil - - -	18,000	10,000
Coal-gas - - -	19,800	11,000

## VOLUMES OF WATER AT DIFFERENT TEMPERATURES (*Kopp*).

Temperature Centigrade.	Volume.	Temperature Centigrade.	Volume.
0	1.	21	1.001776
1	0.999947	22	1.001995
2	0.999908	23	1.002225
3	0.999885	24	1.002465
4	0.999877	25	1.002715
5	0.999883	30	1.004064
6	0.999903	35	1.005697
7	0.999938	40	1.007531
8	0.999986	45	1.009541
9	1.000048	50	1.011766
10	1.000124	55	1.014100
11	1.000213	60	1.016590
12	1.000314	65	1.019302
13	1.000429	70	1.022246
14	1.000556	75	1.025440
15	1.000695	80	1.028581
16	1.000846	85	1.031894
17	1.001010	90	1.035397
18	1.001184	95	1.039094
19	1.001370	100	1.042986
20	1.001567		

## SULPHURIC ACID.

TABLE OF SPECIFIC GRAVITIES, WEIGHTS, AND VOLUMES, AT  
VARIOUS DEGREES TWADDELL.*(Lunge and Isler.)*

Twaddell at 60° Fahr.	Specific Gravity.	100 Parts by Weight contain		1 Cubic Foot of Acid 60° Fahr.		
		SO <sub>3</sub> .	H <sub>2</sub> SO <sub>4</sub> .	Weights Lbs. Avd.	Contains H <sub>2</sub> SO <sub>4</sub> .	Yields Na <sub>2</sub> SO <sub>4</sub> .
					Lbs.	Lbs.
40	1.200	22.30	27.32	74.82	20.44	29.62
41	1.205	22.82	27.95	75.14	21.00	33.43
42	1.210	23.33	28.58	75.45	21.57	31.25
43	1.215	23.84	29.21	75.76	22.14	32.08
44	1.220	24.36	29.84	76.07	22.71	32.90
45	1.225	24.88	30.48	76.38	23.28	33.73
46	1.230	25.39	31.11	76.69	23.85	34.55
47	1.235	25.88	31.70	77.00	24.41	35.37
48	1.240	26.35	32.28	77.32	24.97	36.18
49	1.245	26.83	32.86	77.63	25.54	37.01
50	1.250	27.29	33.43	77.94	26.10	37.82
51	1.255	27.76	34.00	78.25	26.66	38.63
52	1.260	28.22	34.57	78.56	27.23	39.45
53	1.265	28.69	35.14	78.87	27.79	40.27
54	1.270	29.15	35.71	79.19	28.35	41.08
55	1.275	29.62	36.29	79.50	28.92	41.90
56	1.280	30.10	36.87	79.81	29.48	42.72
57	1.285	30.57	37.45	80.12	30.04	43.53
58	1.290	31.04	38.03	80.43	30.60	44.34
59	1.295	31.52	38.61	80.74	31.17	45.16
60	1.300	31.99	39.19	81.06	31.74	45.99
61	1.305	32.46	39.77	81.37	32.32	46.83
62	1.310	32.94	40.35	81.68	32.89	47.65
63	1.315	33.41	40.93	81.99	33.46	48.48
64	1.320	33.88	41.50	82.30	34.03	49.31
65	1.325	34.35	42.08	82.62	34.60	50.13
66	1.330	34.80	42.66	82.93	35.18	50.98
67	1.335	35.27	43.20	83.24	35.79	51.86
68	1.340	35.71	43.74	83.55	36.40	52.74
69	1.345	36.14	44.28	83.86	37.01	53.63
70	1.350	36.58	44.82	84.17	37.63	54.52
71	1.355	37.02	45.35	84.49	38.24	55.41
72	1.360	37.45	45.88	84.80	38.85	56.29
73	1.365	37.89	46.41	85.11	39.46	57.18
74	1.370	38.32	46.94	85.42	40.07	58.05
75	1.375	38.75	47.47	85.73	40.68	58.94
76	1.380	39.18	48.00	86.04	41.29	59.83
77	1.385	39.62	48.53	86.36	41.91	60.72
78	1.390	40.05	49.06	86.67	42.52	61.61
79	1.395	40.48	49.59	86.98	43.13	62.50
80	1.400	40.91	50.11	87.29	43.74	63.38
81	1.405	41.33	50.63	87.60	44.36	64.27
82	1.410	41.76	51.15	87.92	44.97	65.13
83	1.415	42.17	51.66	88.23	45.58	66.02
84	1.420	42.57	52.15	88.54	46.18	66.90
85	1.425	42.96	52.63	88.85	46.78	67.78
86	1.430	43.36	53.11	89.16	47.38	68.65
87	1.435	43.75	53.59	89.47	47.99	69.53

SPECIFIC GRAVITY OF SULPHURIC ACID.

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SULPHURIC ACID—*Continued.*

Twaddell at 60° Fahr.	Specific Gravity.	100 Parts by Weight contain		1 Cubic Foot of Acid 60° Fahr.		
		SO <sub>3</sub> .	H <sub>2</sub> SO <sub>4</sub> .	Weighs Lbs. Avd.	Contains H <sub>2</sub> SO <sub>4</sub> . Lbs.	Yields Na <sub>2</sub> SO <sub>4</sub> . Lbs.
88	1.440	44.14	54.07	89.79	48.59	70.41
89	1.445	44.53	54.55	90.10	49.19	71.28
90	1.450	44.92	55.03	90.41	49.79	72.15
91	1.455	45.31	55.50	90.72	50.39	73.01
92	1.460	45.69	55.97	91.03	50.99	73.88
93	1.465	46.07	56.43	91.35	51.59	74.76
94	1.470	46.45	56.90	91.66	52.19	75.62
95	1.475	46.83	57.37	91.97	52.79	76.49
96	1.480	47.21	57.83	92.28	53.39	77.36
97	1.485	47.57	58.28	92.59	54.00	78.25
98	1.490	47.95	58.74	92.90	54.60	79.12
99	1.495	48.34	59.22	93.22	55.20	79.98
100	1.500	48.73	59.70	93.53	55.84	80.92
101	1.505	49.12	60.18	93.84	56.47	81.82
102	1.510	49.51	60.65	94.15	57.10	82.74
103	1.515	49.89	61.12	94.46	57.73	83.65
104	1.520	50.28	61.59	94.77	58.36	84.56
105	1.525	50.66	62.06	95.09	59.00	85.50
106	1.530	51.04	62.53	95.40	59.62	86.39
107	1.535	51.43	63.00	95.71	60.26	87.32
108	1.540	51.78	63.43	96.02	60.89	88.23
109	1.545	52.12	63.85	96.33	61.52	89.15
110	1.550	52.46	64.26	96.65	62.15	90.06
111	1.555	52.79	64.67	96.96	62.78	90.97
112	1.560	53.12	65.08	97.27	63.42	91.90
113	1.565	53.46	65.49	97.58	64.05	92.81
114	1.570	53.80	65.90	97.89	64.68	93.72
115	1.575	54.13	66.30	98.20	65.31	94.64
116	1.580	54.46	66.71	98.52	65.94	95.54
117	1.585	54.80	67.13	98.83	66.58	96.48
118	1.590	55.18	67.59	99.14	67.21	97.40
119	1.595	55.55	68.05	99.45	67.84	98.30
120	1.600	55.93	68.51	99.76	68.47	99.22
121	1.605	56.30	68.97	100.07	69.10	100.15
122	1.610	56.68	69.43	100.39	69.74	101.05
123	1.615	57.05	69.89	100.70	70.37	101.95
124	1.620	57.40	70.32	101.01	71.07	102.96
125	1.625	57.75	70.74	101.32	71.77	104.00
126	1.630	58.09	71.16	101.64	72.46	105.00
127	1.635	58.43	71.57	101.95	73.16	106.00
128	1.640	58.77	71.99	102.26	73.85	107.00
129	1.645	59.10	72.40	102.57	74.55	108.00
130	1.650	59.45	72.87	102.88	75.25	109.05
131	1.655	59.78	73.23	103.19	75.94	110.04
132	1.660	60.11	73.64	103.50	76.64	111.05
133	1.665	60.46	74.07	103.82	77.33	112.05
134	1.670	60.82	74.51	104.13	78.03	113.05
135	1.675	61.20	74.97	104.44	78.73	114.10
136	1.680	61.57	75.42	104.75	79.42	115.10
137	1.685	61.93	75.86	105.07	80.12	116.10
138	1.690	62.29	76.30	105.38	80.81	117.10
139	1.695	62.64	76.73	105.69	81.51	118.10
140	1.700	63.00	77.17	106.00	82.21	119.15

SULPHURIC ACID—*Continued.*

Twaddell at 60° Fahr.	Specific Gravity.	100 Parts by Weight contain		1 Cubic Foot of Acid 60° Fahr.		
		SO <sub>3</sub> .	H <sub>2</sub> SO <sub>4</sub> .	Weighs Lbs. Avd.	Contains H <sub>2</sub> SO <sub>4</sub> .	Yields Na <sub>2</sub> SO <sub>4</sub> .
					Lbs.	Lbs.
141	1.705	63.35	77.60	106.31	82.90	120.15
142	1.710	63.70	78.04	106.62	83.60	121.15
143	1.715	64.07	78.48	106.94	84.29	122.15
144	1.720	64.43	78.92	107.25	84.99	123.15
145	1.725	64.78	79.36	107.56	85.69	124.20
146	1.730	65.14	79.80	107.87	86.38	125.20
147	1.735	65.50	80.24	108.18	87.08	126.20
148	1.740	65.86	80.68	108.49	87.77	127.20
149	1.745	66.22	81.12	108.80	88.47	128.20
150	1.750	66.58	81.56	109.12	89.17	129.20
151	1.755	66.94	82.00	109.43	89.86	130.20
152	1.760	67.30	82.44	109.74	90.56	131.20
153	1.765	67.65	82.88	110.05	91.25	132.25
154	1.770	68.02	83.32	110.36	91.95	133.25
155	1.775	68.49	83.90	110.68	92.88	134.60
156	1.780	68.98	84.50	110.99	93.81	135.90
157	1.785	69.47	85.10	111.30	94.74	137.30
158	1.790	69.96	85.70	111.61	95.67	138.50
159	1.795	70.45	86.30	111.92	96.60	140.00
160	1.800	70.94	86.90	112.23	97.52	141.30
161	1.805	71.50	87.60	...	...	...
162	1.810	72.08	88.30	...	...	...
163	1.815	72.69	89.05	...	...	...
164	1.820	73.51	90.05	...	...	...
...	1.821	73.63	90.20	...	...	...
...	1.822	73.80	90.40	...	...	...
...	1.823	73.96	90.60	...	...	...
...	1.824	74.12	90.80	...	...	...
165	1.825	74.29	91.00	...	...	...
...	1.826	74.49	91.25	...	...	...
...	1.827	74.69	91.50	...	...	...
...	1.828	74.86	91.70	...	...	...
...	1.829	75.03	91.90	...	...	...
166	1.830	75.19	92.10	...	...	...
...	1.831	75.35	92.30	...	...	...
...	1.832	75.53	92.52	...	...	...
...	1.833	75.72	92.75	...	...	...
...	1.834	75.96	93.05	...	...	...
167	1.835	76.27	93.43	...	...	...
...	1.836	76.57	93.80	...	...	...
...	1.837	76.90	94.20	...	...	...
...	1.838	77.23	94.60	...	...	...
...	1.839	77.55	95.00	...	...	...
168	1.840	78.04	95.60	...	...	...
...	1.840	78.33	95.95	...	...	...
...	1.841	79.19	97.00	...	...	...
...	1.841	79.76	97.70	...	...	...
...	1.841	80.16	98.20	...	...	...
...	1.840	80.57	98.70	...	...	...
...	1.840	80.98	99.20	...	...	...
...	1.839	81.18	99.45	...	...	...
...	1.839	81.39	99.70	...	...	...
...	1.838	81.59	99.95	...	...	...



SPECIFIC GRAVITY AND PERCENTAGE OF CAUSTIC POTASH  
IN AQUEOUS SOLUTION.

Specific Gravity at 15° Cent.	Per-centage of KOH.	Specific Gravity at 15° Cent.	Per-centage of KOH.	Specific Gravity at 15° Cent.	Per-centage of KOH.	Specific Gravity at 15° Cent.	Per-centage of KOH.
1.009	1	1.166	19	1.374	37	1.604	55
1.017	2	1.177	20	1.387	38	1.618	56
1.025	3	1.188	21	1.400	39	1.630	57
1.033	4	1.198	22	1.412	40	1.642	58
1.041	5	1.209	23	1.425	41	1.655	59
1.049	6	1.220	24	1.438	42	1.667	60
1.058	7	1.230	25	1.450	43	1.681	61
1.065	8	1.241	26	1.462	44	1.695	62
1.074	9	1.252	27	1.475	45	1.705	63
1.083	10	1.264	28	1.488	46	1.718	64
1.092	11	1.276	29	1.499	47	1.729	65
1.101	12	1.288	30	1.511	48	1.740	66
1.110	13	1.300	31	1.525	49	1.754	67
1.119	14	1.311	32	1.539	50	1.768	68
1.128	15	1.324	33	1.552	51	1.780	69
1.137	16	1.336	34	1.565	52	1.790	70
1.146	17	1.349	35	1.578	53		
1.155	18	1.361	36	1.590	54		

SPECIFIC GRAVITY AND PERCENTAGE OF CAUSTIC SODA  
IN AQUEOUS SOLUTION.

Specific Gravity at 15° Cent.	Per-centage of NaOH.	Specific Gravity at 15° Cent.	Per-centage of NaOH.	Specific Gravity at 15° Cent.	Per-centage of NaOH.	Specific Gravity at 15° Cent.	Per-centage of NaOH.
1.122	10	1.392	27	1.638	43	1.842	59
1.136	11	1.404	28	1.650	44	1.854	60
1.152	12	1.422	29	1.662	45	1.864	61
1.166	13	1.438	30	1.674	46	1.874	62
1.182	14	1.454	31	1.686	47	1.888	63
1.196	15	1.462	32	1.702	48	1.896	64
1.214	16	1.488	33	1.716	49	1.900	65
1.232	17	1.502	34	1.728	50	1.918	66
1.248	18	1.518	35	1.740	51	1.928	67
1.262	19	1.532	36	1.750	52	1.938	68
1.278	20	1.548	37	1.762	53	1.948	69
1.294	21	1.564	38	1.778	54	1.960	70
1.308	22	1.580	39	1.790	55	1.972	71
1.324	23	1.594	40	1.804	56	1.982	72
1.342	24	1.608	41	1.816	57	1.992	73
1.358	25	1.622	42	1.828	58	2.002	74
1.374	26						

TENSION OF AQUEOUS VAPOUR FOR EACH TENTH OF A DEGREE  
CENTIGRADE FROM 0° TO 30° CENT. (REGNAULT).

Tem- perature ° Cent.	Tension in Mm. of Mercury.	Tem- perature ° Cent.	Tension in Mm. of Mercury.	Tem- perature ° Cent.	Tension in Mm. of Mercury.	Tem- perature ° Cent.	Tension in Mm. of Mercury.
0.0	4.6	4.4	6.3	8.8	8.5	13.2	11.3
0.1	4.6	4.5	6.3	8.9	8.5	13.3	11.4
0.2	4.7	4.6	6.4	9.0	8.6	13.4	11.5
0.3	4.7	4.7	6.4	9.1	8.6	13.5	11.5
0.4	4.7	4.8	6.4	9.2	8.7	13.6	11.6
0.5	4.8	4.9	6.5	9.3	8.7	13.7	11.7
0.6	4.8	5.0	6.5	9.4	8.8	13.8	11.8
0.7	4.8	5.1	6.6	9.5	8.9	13.9	11.8
0.8	4.9	5.2	6.6	9.6	8.9	14.0	11.9
0.9	4.9	5.3	6.7	9.7	9.0	14.1	12.0
1.0	4.9	5.4	6.7	9.8	9.0	14.2	12.1
1.1	5.0	5.5	6.8	9.9	9.1	14.3	12.1
1.2	5.0	5.6	6.8	10.0	9.2	14.4	12.2
1.3	5.0	5.7	6.9	10.1	9.2	14.5	12.3
1.4	5.1	5.8	6.9	10.2	9.3	14.6	12.4
1.5	5.1	5.9	7.0	10.3	9.3	14.7	12.5
1.6	5.2	6.0	7.0	10.4	9.4	14.8	12.5
1.7	5.2	6.1	7.0	10.5	9.5	14.9	12.6
1.8	5.2	6.2	7.1	10.6	9.5	15.0	12.7
1.9	5.3	6.3	7.1	10.7	9.6	15.1	12.8
2.0	5.3	6.4	7.2	10.8	9.7	15.2	12.9
2.1	5.3	6.5	7.2	10.9	9.7	15.3	12.9
2.2	5.4	6.6	7.3	11.0	9.8	15.4	13.0
2.3	5.4	6.7	7.3	11.1	9.9	15.5	13.1
2.4	5.5	6.8	7.4	11.2	9.9	15.6	13.2
2.5	5.5	6.9	7.4	11.3	10.0	15.7	13.3
2.6	5.5	7.0	7.5	11.4	10.1	15.8	13.4
2.7	5.6	7.1	7.5	11.5	10.1	15.9	13.5
2.8	5.6	7.2	7.6	11.6	10.2	16.0	13.5
2.9	5.6	7.3	7.6	11.7	10.3	16.1	13.6
3.0	5.7	7.4	7.7	11.8	10.3	16.2	13.7
3.1	5.7	7.5	7.8	11.9	10.4	16.3	13.8
3.2	5.8	7.6	7.8	12.0	10.5	16.4	13.9
3.3	5.8	7.7	7.9	12.1	10.5	16.5	14.0
3.4	5.8	7.8	7.9	12.2	10.6	16.6	14.1
3.5	5.9	7.9	8.0	12.3	10.7	16.7	14.2
3.6	5.9	8.0	8.0	12.4	10.7	16.8	14.2
3.7	6.0	8.1	8.1	12.5	10.8	16.9	14.3
3.8	6.0	8.2	8.1	12.6	10.9	17.0	14.4
3.9	6.1	8.3	8.2	12.7	10.9	17.1	14.5
4.0	6.1	8.4	8.2	12.8	11.0	17.2	14.6
4.1	6.1	8.5	8.3	12.9	11.1	17.3	14.7
4.2	6.2	8.6	8.3	13.0	11.2	17.4	14.8
4.3	6.2	8.7	8.4	13.1	11.2	17.5	14.9

TENSION OF AQUEOUS VAPOUR—*Continued.*

Tem- perature ° Cent.	Tension in Mm. of Mercury.	Tem- perature ° Cent.	Tension in Mm. of Mercury.	Tem- perature ° Cent.	Tension in Mm. of Mercury.	Tem- perature ° Cent.	Tension in Mm. of Mercury.
17.6	15.0	20.8	18.3	23.9	22.1	27.0	26.5
17.7	15.1	20.9	18.4	24.0	22.2	27.1	26.7
17.8	15.2	21.0	18.5	24.1	22.3	27.2	26.8
17.9	15.3	21.1	18.6	24.2	22.5	27.3	27.0
18.0	15.4	21.2	18.7	24.3	22.6	27.4	27.1
18.1	15.5	21.3	18.8	24.4	22.7	27.5	27.3
18.2	15.6	21.4	19.0	24.5	22.9	27.6	27.5
18.3	15.7	21.5	19.1	24.6	23.0	27.7	27.6
18.4	15.8	21.6	19.2	24.7	23.1	27.8	27.8
18.6	15.9	21.7	19.3	24.8	23.3	27.9	27.9
18.7	16.0	21.8	19.4	24.9	23.4	28.0	28.1
18.8	16.1	21.9	19.5	25.0	23.5	28.1	28.3
18.9	16.2	22.0	19.7	25.1	23.7	28.2	28.4
19.0	16.3	22.1	19.8	25.2	23.8	28.3	28.6
19.1	16.4	22.2	19.9	25.3	24.0	28.4	28.8
19.2	16.6	22.3	20.0	25.4	24.1	28.5	28.9
19.3	16.7	22.4	20.1	25.5	24.3	28.6	29.1
19.4	16.8	22.5	20.3	25.6	24.4	28.7	29.3
19.5	16.9	22.6	20.4	25.7	24.6	28.8	29.4
19.6	17.0	22.7	20.5	25.8	24.7	28.9	29.6
19.7	17.1	22.8	20.6	25.9	24.8	29.0	29.8
19.8	17.2	22.9	20.8	26.0	25.0	29.1	30.0
19.9	17.3	23.0	20.9	26.1	25.1	29.2	30.1
20.0	17.4	23.1	20.0	26.2	25.3	29.3	30.3
20.1	17.5	23.2	21.1	26.3	25.4	29.4	30.5
20.2	17.6	23.3	21.3	26.4	25.6	29.5	30.7
20.3	17.7	23.4	21.4	26.5	25.7	29.6	30.8
20.4	17.8	23.5	21.5	26.6	25.9	29.7	31.0
20.5	17.9	23.6	21.7	26.7	26.0	29.8	31.2
20.6	18.0	23.7	21.8	26.8	26.2	29.9	31.4
20.7	18.2	23.8	21.9	26.9	26.4		

*Example:—*

Find the amount of aqueous vapour in gas at 50° Fahr. (10° Cent.).

Find the tension in mm. of mercury by table, divide by 760 mm. and you have the percentage of aqueous vapour; multiply by 100 = grains per 100 cub. ft.

Find weight of a cubic foot of aqueous vapour =  $H_2O = 18 \div 2 = 9 \times 37 = 333.760$  9.200 (1.20 per cent.

1,000,000 cub. ft. of gas  $\times 1.2 = 1,200,000$ .

$1,200,000 \times 12 \div 70,000 = 57.08$  gallons aqueous vapour per million cub. ft.

## LOSS OF ILLUMINATING POWER BY ADMIXTURE OF AIR.

*(Dr E. G. Lowe.)*

Percentage of Air in Mixture.	Loss of Illuminating Power.	Loss of Illuminating Power for 1 per cent. Air.	Percentage of Air in Mixture.	Loss of Illuminating Power.	Loss of Illuminating Power for 1 per cent. Air.
	Per cent.	Per cent.		Per cent.	Per cent.
2.82	5.72	2.03	17.65	44.30	2.51
4.94	10.08	2.04	19.84	51.19	2.58
5.40	11.24	2.08	21.56	56.69	2.63
8.51	18.04	2.12	22.22	58.88	2.65
8.95	19.06	2.13	24.16	64.51	2.67
9.62	21.16	2.20	27.69	72.82	2.63
10.40	23.24	2.24	31.30	80.44	2.57
11.20	26.66	2.38	32.95	83.36	2.53
12.35	29.02	2.35	34.53	87.02	2.52
12.81	30.48	2.38	37.50	90.37	2.41
15.25	37.66	2.47	40.79	93.82	2.30
16.98	42.28	2.49	...	...	...

Experiments carried out on 25-27 candle water-gas with a Bray's Slit Union No. 7 Burner, with a regulated consumption of 5 cub. ft. per hour.

## MAXIMUM VAPOUR PRESSURES OF NAPHTHALENE.

*(By W. R. Allan, M.A.)*

Temperature ° Fahr.	Corresponding Vapour Pressure.	Weight of Naphthalene Saturating 100 cub. ft. of Gas.	Temperature ° Fahr.	Corresponding Vapour Pressure.	Weight of Naphthalene Saturating 100 cub. ft. of Gas.
	Mm.	Grains.		Mm.	Grains.
32	0.022	6.0	158	3.95	1050.0
41	0.034	9.8	167	5.43	1430.0
50	0.047	14.1	176	7.4	1898.0
59	0.062	19.0	185	9.8	2426.0
68	0.080	24.6	194	12.6	3035.0
77	0.103	30.9	203	15.5	3727.0
86	0.135	39.5	212	18.5	4420.0
95	0.210	57.5	221	22.4	...
104	0.320	83.5	230	27.3	...
113	0.510	132.0	239	32.4	...
122	0.810	208.0	248	40.2	...
131	1.260	317.0	257	49.8	...
140	1.830	482.0	266	61.9	...
149	2.650	723.0	...	...	...

# SOLUBILITY OF GASES IN WATER.

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SOLUBILITY OF GASES IN WATER (BY VOLUME) AT A PRESSURE OF 760 MM. (29.92 INS.). (*Bunsen.*)

1 Vol. of Water Dissolves at ° Cent.	Am- monia.	Atmos- pheric Air.	Carbon Dioxide.	Carbon Monoxide.	Hydro- gen.	Nitrogen.	Oxygen.	Sulphur- etted Hydro- gen.
0°	1049.6	0.02471	1.7967	0.03287	0.0193	0.02035	0.04114	4.3706
1°	1020.8	0.02406	1.7207	0.03207	0.0193	0.01981	0.04007	4.2874
2°	993.3	0.02345	1.6481	0.03131	0.0193	0.01932	0.03907	4.2053
3°	967.0	0.02287	1.5787	0.03057	0.0193	0.01884	0.03810	4.1243
4°	941.9	0.02237	1.5126	0.02987	0.0193	0.01838	0.03717	4.0442
5°	917.9	0.02179	1.4497	0.02920	0.0193	0.01794	0.03628	3.9652
6°	895.0	0.02128	1.3901	0.02857	0.0193	0.01752	0.03554	3.8872
7°	873.1	0.02080	1.3339	0.02796	0.0193	0.01713	0.03465	3.8103
8°	852.1	0.02034	1.2809	0.02739	0.0193	0.01675	0.03389	3.7345
9°	832.0	0.01992	1.2311	0.02686	0.0193	0.01640	0.03317	3.6596
10°	812.8	0.01953	1.1847	0.02635	0.0193	0.01607	0.03250	3.5858
11°	794.3	0.01916	1.1416	0.02588	0.0193	0.01577	0.03189	3.5132
12°	776.6	0.01882	1.1018	0.02544	0.0193	0.01549	0.03133	3.4415
13°	759.6	0.01851	1.0653	0.02504	0.0193	0.01523	0.03082	3.3708
14°	743.1	0.01822	1.0321	0.02466	0.0193	0.01500	0.03034	3.3012
15°	727.2	0.01795	1.0020	0.02432	0.0193	0.01478	0.02989	3.2326
16°	711.8	0.01771	0.9753	0.02402	0.0193	0.01458	0.02949	3.1651
17°	696.9	0.01750	0.9519	0.02374	0.0193	0.01441	0.02914	3.0986
18°	682.3	0.01732	0.9318	0.02350	0.0193	0.01426	0.02884	3.0331
19°	668.0	0.01717	0.9150	0.02329	0.0193	0.01423	0.02858	2.9687
20°	654.0	0.01704	0.9014	0.02312	0.0193	0.01403	0.02838	2.9053

## ATOMIC WEIGHTS.

	Sym- bol.	Weight.		Sym- bol.	Weight.
Aluminium	-	Al 27.1	Fluorine	-	F 19.00
Antimony	-	Sb 120.00	Gallium	-	Ga 70.00
Argon (?)	-	A 40.00	Germanium	-	Ge 72.00
Arsenic	-	As 75.00	Gold	-	Au 197.2
Barium	-	Ba 137.4	Helium (?)	-	He 4.00
Beryllium	-	Be 9.1	Hydrogen	-	H 1.01
Bismuth	-	Bi 208.5	Indium	-	In 114.00
Boron	-	B 11.00	Iodine	-	I 126.85
Bromine	-	Br 79.96	Iridium	-	Ir 193.00
Cadmium	-	Cd 112.00	Iron	-	Fe 56.00
Cæsium	-	Cs 113.00	Lanthanium	-	La 138.00
Calcium	-	Ca 40.00	Lead	-	Pb 206.9
Carbon	-	C 12.00	Lithium	-	Li 7.03
Cerium	-	Ce 140.00	Magnesium	-	Mg 24.36
Chlorine	-	Cl 35.45	Manganese	-	Mn 55.00
Chromium	-	Cr 52.1	Mercury	-	Hg 200.3
Cobalt	-	Co 59.00	Molybdenum	-	Mo 96.00
Copper	-	Cu 63.6	Neodidymium (?)	-	Nd 144.00
Erbium (?)	-	Er 166.00	Nickel	-	Ni 58.7

ATOMIC WEIGHTS—*Continued.*

	Sym- bol.	Weight.		Sym- bol.	Weight.
Niobium - - -	Nb	94.00	Sodium - - -	Na	23.05
Nitrogen - - -	N	14.04	Strontium - - -	Sr	87.6
Osmium - - -	Os	191.00	Sulphur - - -	S	32.06
Oxygen - - -	O	16.00	Tantalum - - -	Ta	183.00
Palladium - - -	Pd	106.00	Tellurium - - -	Te	127.00
Phosphorus - - -	P	31.0	Thallium - - -	Tl	204.1
Platinum - - -	Pt	194.8	Thorium - - -	Th	232.00
Potassium - - -	K	39.15	Titanium - - -	Ti	48.1
Praseodidymium (?) -	Pr	140.00	Tin - - -	Sn	118.5
Rhodium - - -	Rh	103.00	Tungsten - - -	W	184.00
Rubidium - - -	Rb	85.4	Uranium - - -	U	239.5
Ruthenium - - -	Ru	101.7	Vanadium - - -	V	57.2
Samarium (?) - - -	Sa	150.00	Ytterbium - - -	Vb	173.00
Scandium - - -	Sc	44.1	Yttrium - - -	Y	89.00
Selenium - - -	Se	79.1	Zinc - - -	Zn	65.4
Silicium - - -	Si	28.4	Zirconium - - -	Zr	90.6
Silver - - -	Ag	107.93			

PERCENTAGE BY VOLUME, CORRESPONDING TO THE WEIGHT  
IN GRAINS OF CO<sub>2</sub> PER CUB. FT. OF GAS, AT  
60° FAHR. AND 30-IN. BAR.

Percentage by Volume.	Grains CO <sub>2</sub> per Cub. Ft.	Percentage by Volume.	Grains CO <sub>2</sub> per Cub. Ft.	Percentage by Volume.	Grains CO <sub>2</sub> per Cub. Ft.
0.1	0.817	1.1	8.987	2.1	17.157
0.2	1.634	1.2	9.807	2.2	17.974
0.3	2.451	1.3	10.621	2.3	18.791
0.4	3.268	1.4	11.438	2.4	19.608
0.5	4.085	1.5	12.255	2.5	20.425
0.6	4.902	1.6	13.072	2.6	21.242
0.7	5.719	1.7	13.889	2.7	22.059
0.8	6.536	1.8	14.706	2.8	22.876
0.9	7.353	1.9	15.523	2.9	23.693
1.0	8.170	2.0	16.340	3.0	24.510

PERCENTAGE BY VOLUME, CORRESPONDING TO THE WEIGHT  
IN GRAINS OF  $\text{SH}_2$  PER CUB. FT. OF GAS, AT  
60° FAHR. AND 30-IN. BAR.

Percentage by Volume.	Grains $\text{SH}_2$ per Cub. Ft.	Percentage by Volume.	Grains $\text{SH}_2$ per Cub. Ft.	Percentage by Volume.	Grains $\text{SH}_2$ per Cub. Ft.
0.1	0.63	1.1	6.94	2.1	13.25
0.2	1.26	1.2	7.57	2.2	13.88
0.3	1.89	1.3	8.20	2.3	14.51
0.4	2.52	1.4	8.83	2.4	15.14
0.5	3.15	1.5	9.46	2.5	15.77
0.6	3.78	1.6	10.09	2.6	16.40
0.7	4.41	1.7	10.72	2.7	17.03
0.8	5.04	1.8	11.35	2.8	17.66
0.9	5.67	1.9	11.98	2.9	18.30
1.0	6.31	2.0	12.62	3.0	18.93

PERCENTAGE BY VOLUME, CORRESPONDING TO THE WEIGHT  
IN GRAINS OF  $\text{NH}_3$  PER CUB. FT. OF GAS, AT  
60° FAHR. AND 30-IN. BAR.

Percentage by Volume.	Grains $\text{NH}_3$ per Cub. Ft.	Percentage by Volume.	Grains $\text{NH}_3$ per Cub. Ft.	Percentage by Volume.	Grains $\text{NH}_3$ per Cub. Ft.
0.1	0.315	1.1	3.471	2.1	6.627
0.2	0.631	1.2	3.787	2.2	6.943
0.3	0.946	1.3	4.102	2.3	7.258
0.4	1.262	1.4	4.418	2.4	7.574
0.5	1.578	1.5	4.734	2.5	7.890
0.6	1.893	1.6	5.049	2.6	8.205
0.7	2.208	1.7	5.365	2.7	8.521
0.8	2.524	1.8	5.680	2.8	8.836
0.9	2.840	1.9	5.996	2.9	9.152
1.0	3.156	2.0	6.312	3.0	9.468

## NOTES ON CALORIFIC VALUE.

A gram-calorie is the amount of heat required to raise 1 gram of water 1° Centigrade.

A kilo-calorie is the amount of heat required to raise 1 kilogramme of water (1 litre) 1° Centigrade.

A British Thermal Unit (B.Th.U.) is the amount of heat required to raise 1 lb. of water 1° Fahrenheit.

Calories per cubic foot are the number of kilogrammes of water (litres) raised through 1° Cent. by complete combustion of 1 cubic foot of gas at N.T.P.

Calories per cubic metre are the number of kilogrammes of water (litres) raised through 1° Cent. by complete combustion of 1 cubic metre of gas.

B.Th.U.'s per cubic foot are the number of lbs. of water which can be raised through 1° Fahr. by complete combustion of 1 cubic foot of gas.

To convert calories per cubic foot into B.Th.U.'s per cubic foot, multiply by 3.968 (3.97).

To convert calories per cubic foot into calories per cubic metre, multiply by 35.316.

To convert B.Th.U.'s per cubic foot into calories per cubic metre, multiply by 8.9.

The calorific value of 1 lb. of fuel expressed in B.Th.U. is the Fahrenheit-lb. unit per lb.

The amount of water which 1 lb. of fuel will evaporate, or the evaporative power per lb., is obtained from the calorific value of 1 lb. of fuel expressed in B.Th.U. divided by 967 (latent heat of steam in degrees Fahr.).

If a Centigrade thermometer is used, and it is desired to arrive at the evaporative value per lb., divide by 537 (latent heat of steam in degrees Cent.) instead of by 967.

### *Example:—*

1 lb. of fuel has a value of 11,800 B.Th.U.'s per lb.,  
 then 1 gram of fuel has a value of  $11,800 \times \frac{1}{2.205}$  (6555.5) calories per gram;  
 or 1 lb. of fuel has a value of  $11,800 \times \frac{1}{2.205}$  (6555.5) calories per lb.;  
 or 1 ton of fuel has a value of  $11,800 \times \frac{2240}{2.205}$  (6555.5) calories per ton, &c.

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